

FINAL REPORT

Demonstrate a Low Biochemical Oxygen Demand Aircraft Deicing Fluid

ESTCP Project WP-200905

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14. ABSTRACT

Aircraft deicing fluids are required to remove frozen precipitation from aircraft prior to flight, ensuring mission capability in winter conditions. The primary component in conventional fluids is propylene glycol (PG), which can exhibit a high biochemical oxygen demand (BOD) when it degrades. The aim of this demonstration was to investigate whether one reduced PG aircraft deicing fluid, EcoFlo (and later EcoFlo II), was effective at deicing aircraft while having no negative effects on flight performance, operational safety and aircraft materials. Evaluation included laboratory testing for compatibility with military aircraft materials, wind tunnel testing for fluid residue concerns (blurring windows/optical ports and leaving slippery surfaces), and an aircraft demonstration for effective and safe removal of frozen precipitation. The demonstration indicated that residue issues are still an obstruction to implementation and further reformulation and improvement must be considered.

15. SUBJECT TERMS

Aircraft Deicing Fluid, ADF, EcoFlo, EcoFlo II, propylene glycol, low biochemical oxygen demand, environmentally friendly deicing fluid

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LIST OF ACRONYMS

ADF Aircraft Deicing Fluid

AFB Air Force Base

AMCTES Air Mobility Command Test and Evaluation Squadron

AMS Aerospace Material Specification

ANG Air National Guard

BOD Biochemical Oxygen Demand
BRAC Base Realignment and Closure
COD Chemical Oxygen Demand

CTC Concurrent Technologies Corporation

D³ Degradable by Design Deicer™

DI Deionized

DoD Department of Defense

DSC Differential Scanning Calorimetry
EPA Environmental Protection Agency

EPCRA Emergency Planning and Community Right-To-Know Act ESTCP Environmental Security Technology Certification Program

HE Hydrogen Embrittlement

LO Low Observable

LOUT Lowest Operational Use Temperature NFARS Niagara Falls Air Reserve Station

NPDES National Pollutant Discharge Elimination System

NPE Nonylphenol Ethoxylate

NSPS New Source Performance Standards

OA Operational Assessment

PG Propylene Glycol

PMC Polymer Matrix Composite
SAE Society of Aerospace Engineers

SERDP Strategic Environmental Research and Development Program

SME Subject Matter Experts

WSET Water Spray Endurance Time

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EXECUTIVE SUMMARY

Aircraft deicing fluids are required to remove frozen precipitation from aircraft prior to flight, ensuring mission capability in winter conditions. Without effective removal of frozen precipitation, lift and control might be compromised and safety of flight is jeopardized. A problem with conventional fluids in use today, however, is that the primary component is propylene glycol (PG), which can exhibit a high biochemical oxygen demand (BOD) when it degrades. Thus the waste fluid can either threaten aquatic life or impede the effectiveness of waste water treatment processes, depending on where runoff from airfield deicing operations is directed. Some newer deicing fluid formulations target the reduction or elimination of PG to assuage the BOD impact and reduce related runoff handling and permitting costs.

The aim of this demonstration was to investigate whether one reduced PG aircraft deicing fluid, EcoFlo (and later EcoFlo II), was effective at deicing aircraft while having no negative effects on flight performance, operational safety and aircraft materials. EcoFlo and EcoFlo II had both been tested for compliance with SAE AMS 1424G, the specification covering aircraft deicing fluids, and EcoFlo had been marketed for commercial aircraft use.

Prior to the demonstration involving application of EcoFlo on operational aircraft, two laboratory evaluations were performed. The first consisted of testing the compatibility of EcoFlo with materials likely to be found on military aircraft but not commercial aircraft (and so, not covered by SAE AMS 1424G). For most of the materials tested, EcoFlo was shown to either have no impact or no impact more significant than that of the baseline fluid, a conventional PG fluid. The few cases where EcoFlo did not perform as anticipated were identified for future evaluation should the fluid be considered for aircraft featuring those specific materials.

The second laboratory evaluation involved testing in a wind tunnel. As some previous reduced PG deicing fluids had shown the tendency to leave a residue that both obscured visibility through windows or observation ports and made surfaces excessively slippery and hindered post flight inspection and maintenance, the project team determined that a wind tunnel evaluation might be merited prior to investing the time and effort in a full aircraft demonstration. Surfaces exposed to the fluid were submitted to airflow consistent with takeoff velocities and then tested for impeded visibility and slipperiness. Although this evaluation was a simple approximation and could not duplicate the complex airflows encountered by various parts of an aircraft, it provided some indication that EcoFlo was likely to act similarly to conventional PG fluids and not leave a significant residue.

Prior to the full demonstration, the manufacturer of EcoFlo informed the project team that they were planning to market a new formulation, EcoFlo II (containing more PG, but still featuring a lower BOD than conventional PG fluids) and eventually discontinue EcoFlo. The project team considered the limited information available on the proprietary formulation and determined that

it was unlikely that EcoFlo II would perform worse than EcoFlo in any of the laboratory evaluations already completed. So EcoFlo II was acquired for the full scale demonstration.

The full demonstration was carried out at Joint Base McGuire-Dix-Lakehurst on 9 February 2012. Maintenance personnel evaluated EcoFlo II for deicing effectiveness, including time, quantity of fluid, and labor required to thoroughly remove frozen precipitation on KC-135 aircraft. For comparison, a second aircraft was deiced with conventional PG fluid. A test flight crew checked for any inflight performance impacts attributable to the fluid, and then the maintenance crew performed post flight evaluations.

The experienced maintenance crew observed that the EcoFlo effectively deiced the aircraft in a time frame and using a quantity of fluid typical for that type of aircraft with that quantity of frozen precipitation, but due to rapidly changing weather conditions, a one-to-one, quantitative comparison to the conventional PG operation was inconclusive (the PG operation was likely aided by radiant heat when the skies cleared).

Flight characteristics were not impacted after the aircraft was deiced, but windows and viewing ports were obscured by fluid residue. Also, after the flight, aircraft surfaces were observed to be extremely slippery and a fall hazard for post flight inspection and maintenance. These factors led the onsite evaluation team to discontinue any further application of fluid (effectively ending the demonstration) and conclude that the EcoFlo II was not suitable for deicing on KC-135 aircraft.

EcoFlo and EcoFlo II were formulated to reduce BOD impact while not affecting aircraft flight and maintenance operations (i.e., by not leaving any slippery, blurry residue), but the fluids still showed these negative effects during this specific event. If this was an anomaly, the factors leading to this unexpected performance must be understood and controlled, otherwise the fluid must be reformulated to reliably prevent residue issues before implementation can be considered.

1.0 INTRODUCTION

1.1 BACKGROUND

The Air Force claims to have an all-weather flying capability but many times missions are placed at risk by weather conditions. During icy and snowy weather the aerodynamic surfaces of the aircraft must be free of ice and snow to achieve proper aerodynamic lift. Freeing the aerodynamic surfaces of frozen contamination is accomplished by spraying a heated fluid on the surfaces which must melt and/or debond the ice or snow. After the fluid loses it heat, it must demonstrate that it will not refreeze on the aircraft.

Aircraft deicing fluid (ADF) runoff is a significant environmental problem at airports. The discharge of ADF into bases' storm water management systems is subject to permitting and reporting requirements under the National Pollutant Discharge Elimination System (NPDES) program and the Emergency Planning and Community Right-To-Know Act (EPCRA). Also, new sources must consider New Source Performance Standards (NSPS) promulgated by the EPA under 40 CFR Part 449 in 2012. The Department of Defense (DoD) has made the reduction or elimination of the use of propylene glycol an environmental priority to help manage permitting under NPDES. Because each airfield is unique and storm water discharge permits are negotiated on a case-by-case basis by state environmental agencies some bases face more stringent regulation than others.

The present ADF of choice by the United States Air Force is propylene glycol (PG), which has a relatively high biochemical oxygen demand (BOD) and chemical oxygen demand (COD) and can deplete oxygen levels in receiving waters and threaten oxygen dependent aquatic life. Also, high concentrations of deicing fluids are known to cause acute aquatic toxicological effects, due mainly to additives (e.g., to improve corrosion inhibition) and not the PG itself. Per AMS 1424G, Deicing/Anti-icing Fluid, Aircraft, SAE Type I [1], the SAE G-12 Aircraft Ground Deicing Committee, in accordance with EPA permitting requirements, has established a toxicity limit of 4,000 mg/L (at an ADF concentration that provides a -26°C freezing point) for all Type I (deicing type) fluids, regardless of the freezing point depressant (e.g., propylene glycol, ethylene glycol, and polyol based fluids).

A product, developed by Battelle Memorial Institute using SERDP funding, exhibits the promise to be more environmentally friendly and cost effective than PG. The product was originally named Degradable by Design DeicerTM (D^3). The product failed field demonstrations due to visibility degradation [through aircraft windows] and slipperiness. The fluid was subsequently reformulated to eliminate these negative effects. It has been licensed to Octagon Process LLC (Octagon Process has been purchased by Clariant Corporation) under the product name EcoFlo.

This program involved laboratory evaluations of an EcoFlo product followed by a field demonstration for ice removal and prevention of ice formation. The full demonstration was carried out on a KC-135 Aircraft supplied by the 108th Wing, New Jersey Air National Guard, located on Joint Base McGuire-Dix-Lakehurst.

1.2 OBJECTIVE OF THE DEMONSTRATION

The objective of the demonstration was to collect operational and performance data to demonstrate that this bio-based, reduced propylene glycol, Type I ADF is an acceptable replacement for the current conventional PG ADF. This fluid will significantly reduce the utilization of those hazardous materials listed in Table 1.

Specifically, the test objectives were as follows:

- 1. Illustrate the effectiveness of the ADF as an operationally suitable deicing fluid. The fluid should have left insignificant residue, comparable to that of PG, and should have demonstrated equal or less visual degradation when compared to PG.
- 2. Identify any residual characteristics of the ADF during and following a successful operational flight after application of the fluid. The team inspected for residue remaining on the aircraft, leading edge dryness, fluid shearing and migration, and streaking.
- 3. Determine the operational benefits and/or potential issues associated with use of the ADF by a facility. The base observers and flight crews were asked: "Is there any noticeable difference in the handling of the aircraft? Is the material compatible with present spraying equipment and base deicing operations? Will Base Operations recommend use of the product?"
- 4. Determine cost benefits of adopting the alternative ADF. Additionally, using a previously developed template under ESTCP project WP-200409, determine the environmental cost impact on the base if this fluid were accepted for use.
- 5. Conduct material compatibility testing.

Table 1 Target Hazardous Material Summary

Target Material	Current Process	Applications	Current Specifications	Affected Programs	Candidate Parts and Substrates
Propylene Glycol	Aircraft Deicing Fluid	Heat transfer, Abrasion, and Freezing Point Depression During Aircraft Deicing	SAE AMS 1424	All Aircraft	Aerospace Materials

Additive Packages (Proprietary chemicals,	Aircraft Deicing Fluid	Corrosion Inhibition	SAE AMS 1424	All Aircraft	Aerospace Materials
e.g., 4-, and 5-, methylbenzotriazole, Glycols, Triazoles, Diethonlonie)					

1.3 REGULATORY DRIVERS

Aircraft deicing fluid runoff is covered by the National Pollution Discharge Elimination System (NPDES) authorized by the Clean Water Act. Additionally, new sources of ADF may be impacted by New Source Performance Standards (NSPS) promulgated by the EPA under 40 CFR Part 449 in 2012.

2.0 DEMONSTRATION TECHNOLOGY

2.1 TECHNOLOGY DESCRIPTION

The basic requirement of a deicing fluid is to transfer heat to frozen aircraft surfaces to either melt the frost, or de-bond the frozen ice or snow allowing it to run off the aircraft leaving the surface free of frost, ice and snow. The fluid is heated to approximately 180°F to increase the melting/ de-bonding effectiveness. Also, pressurized application of the fluid provides mechanical force to abrade and dislodge frozen substances. It is critical that the fluid contributes to freezing point depression when mixed with the melted contamination so that nothing will refreeze on the aircraft. A picture of a typical deicing operation can be seen in Figure 1.



Figure 1 Typical Deicing Operation

For alternatives the fluid should have a viscosity that will allow pumping to occur without changing existing aircraft deicing equipment, and constituents that do not harm hoses and seals. The fluid must also be compatible with unique military materials.

The specific technologies evaluated, EcoFlo and EcoFlo II aircraft deicing fluids, are SAE AMS 1424 compliant alternative to conventional PG fluids, developed by Battelle and manufactured by Clariant Corporation. Both formulations of EcoFlo have a lower BOD and COD than conventional PG fluids. The fluid also exhibits reduced aquatic toxicity characteristics as it includes no triazoles (such as 5-methyl-1H-benzotriazole or 4-methyl-1H-benzotriazole) and no nonylphenol ethoxylate (NPE) surfactants. Triazoles have historically been added to deicing fluids as a part of the corrosion inhibition package. They are identified as possible carcinogens and induce toxic responses in aquatic plants and animals. NPE surfactants are used in deicing fluids as wetting agents which increase the surface activity of the fluid. The EPA has asked chemical manufacturers to voluntarily phase the chemical out of their products as NPEs and their decomposition products can harm aquatic plant and animal species.

When this effort was initiated, only the EcoFlo formulation was being marketed by the manufacturer. Exact information on constituents and concentrations was considered proprietary

and not shared, but characteristics and toxicity, as required for qualification to SAE AMS 1424, were available. The EcoFlo formulation was provided by the manufacturer for the material compatibility testing and the wind tunnel testing.

Subsequent to the laboratory testing, and prior to the aircraft demonstration, a new formulation called EcoFlo II was developed. Clariant Corporation informed the project team that they would likely discontinue EcoFlo in favor of EcoFlo II. Clariant provided no detailed information on the reformulation beyond stating that they reduced the volume fraction of glycerin relative to PG to reduce the viscosity and increase the freezing point at higher fluid concentrations.

Given budget and schedule constraints, the project team acknowledged that repeating laboratory testing for the new formulation would be unfeasible. The team considered that EcoFlo II had passed SAE AMS 1424 testing and, assuming fluid performance varied somewhat linearly with constituent quantities and that the concentration of PG was somewhere between that of EcoFlo and Octaflo EF (a conventional PG-based deicer), the performance should be no worse than that of the original EcoFlo. It was determined by EcoFlo project team Subject Matter Experts (SME) that the reformulation would not significantly impact material compatibility properties and the demonstration moved forward with EcoFlo II. Table 2 lists SAE AMS 1424 evaluation results (including COD and freezing points at various concentrations) of EcoFlo, EcoFlo II and Octaflo EF.

Table 2 EcoFlo/EcoFlo II/PG Characteristics

Property	EcoFlo	EcoFlo II	Octaflo EF
Corrosion	Values of 1	Values of 1	Pass
HE	Passed	Passed	Pass
COD (neat)	1.20	1.29	1.59
Viscosity (6 RPM)			
• +20°C	65	66	30
• 0°C	220	178	140
• -10°C	500	363	300
• -20°C	1500	911	700
Freezing Point			
30:70	-10°C	-10°C	-11°C
40:60	-18°C	-15°C	-19°C
45:55	-22°C	-21°C	-22°C
50:50	-26°C	-32°C	-28°C
55:45	-33°C	-37°C	-34°C
60:40	-39°C	-42°C	-40°C
65:35	-42°C		-54°C
WSET			
• 50/50	5 min 26 s	5 min 00 s	5 min 13 s
• 65/35	7 min 06 s	6 min 45 s	6 min 11 s

(All concentrations are ADF concentrate/water, by %volume)

Table 2 Notes:

The hydrogen embrittlement (HE) evaluation ensures that the fluid does not contribute to hydrogen absorption by high strength steels.

Water spray endurance time (WSET) represents the ability of the fluid to prevent ice formation when exposed to a water spray for a short time period (3 minutes minimum, per SAE AMS 1424).

2.2 TECHNOLOGY DEVELOPMENT

This project follows previously completed Battelle D³ ESTCP aircraft deicing fluid field demonstration projects led by ASC/ENVV (312 AESG/ENF), WP-200124 and WP-200409. Events leading up to this demonstration are as follows:

1998-2000 (Pre-ESTCP):

Concept generation and laboratory testing occurred under Battelle funding, giving Battelle an intellectual property (IP) position.

2001-2003 (ESTCP):

A ready-to-use ADF (D³ 1036) was prepared, based on Battelle's background IP, and certification testing was performed (using Battelle funding).

ADF performance was demonstrated on man-made ice and snow under controlled conditions in the McKinley Climatic Chamber at Eglin AFB (April 2002).

Performance of the ADF formulation was demonstrated, with flight testing, at the Niagara Falls Air Reserve Station (NFARS).

2004-2006 (ESTCP):

A reformulation (D³ 1216D) was developed and certified by Battelle.

Spray test demonstrations of the fluid were performed.

The ADF was again reformulated as a low-foam, concentrate ADF (D³ 1705) and certification was attempted (under Battelle funding). The fluid did not pass the hydrogen embrittlement portion of AMS 1424E and could not be certified, therefore the planned demo/flight test at Niagara Falls Air Reserve Station (NFARS) was cancelled.

2007-2009 (ESTCP, Commercial Funding):

The final formulation of D³ fluid failed residue testing and visual degradation during the demonstration.

The fluid was reformulated again, incorporating additional propylene glycol, passed SAE AMS 1424, and was licensed to Octagon Corporation and marketed as EcoFlo.

2009-2012 (ESTCP):

Octagon requested one more demonstration opportunity for Air Force acceptance, and this project was initiated. During the preparations for the demonstration, Octagon LLC was purchased by Clariant Corporation. Also during this time EcoFlo was reformulated, based on performance of the commercial ADF to date. The new formulation, EcoFlo II, contains a

higher proportion of PG, and was approved for use in this demonstration with the understanding that the manufacturer intended to discontinue production of the original EcoFlo.

2.3 ADVANTAGES AND LIMITATIONS OF THE TECHNOLOGY

EcoFlo is a hybrid fluid which contains approximately 50% propylene glycol (exact concentrations are considered proprietary by the manufacturer and not shared). The main advantages of EcoFlo, compared to existing PG-based ADF, include

- Reduced oxygen demand for biodegradation
- Reduced toxicity
- Reduced odor associated with degradation
- Anticipated lower life-cycle deicing costs.
- Reduction on the use of PG
- Reduction, through utilization, of a waste product that comes from bio-fuel production.

Limitations of EcoFlo are higher viscosity, reduced freezing point depression capabilities and higher surface tension than PG. These are not seen as major disadvantages, but do denote the physical chemistry differences between EcoFlo and pure PG. The most significant limitation is the lowest operational use temperature (LOUT), which is -33°C for PG and -30.5°C for EcoFlo. Also the higher surface tension and higher viscosity of EcoFlo does raise concerns over the ADF leaving residue on the aircraft after deicing operations. The reformulated version, EcoFlo II, claims a lower freezing point and lower viscosity.

3.0 PERFORMANCE OBJECTIVES

The goal of this evaluation was to determine if EcoFlo II performs as well as or better than PG-based deicing fluids. The demonstration included evaluation of the performance objectives captured in the Tables 2 and 3 below, with a discussion of key objectives following each table.

Table 3 Qualitative Performance Objectives

Performance Objective	Data Requirements	Success Criteria	Results
Fluid is effective in removing snow and ice from aircraft	Observations collected from personnel with experience in aircraft deicing operations: flight line personnel, deicing truck operator, flight crew, various DoD and contractor personnel. Observation/Data sheets will be collected for each deicing event.	Concurrence among stakeholders that fluid is effective	Fluid did effectively remove frozen contamination PASS
Fluid coats the aircraft surface in a smooth and consistent manner with no foam. Fluid has good wetting characteristics and exhibits no fish eyes (indicating and oil/water-like mix).	Observations collected from personnel with experience in aircraft deicing operations: flight line personnel, deicing truck operator, flight crew, various DoD and contractor personnel. Observation/Data sheets will be collected for each deicing event.	Does not form persistent foam on deiced surfaces, i.e., foam that does not rapidly collapse or causes the surface to have the appearance of snow or slush. ADF show good wetting without film breaks, crawling, or fish eyes.	Fluid was observed to exhibit some foaming which dissipated rapidly. Fluid appeared to flow and wet the surface adequately PASS
Fluid is substantially removed from the plane surface during takeoff and flight, in a manner similar to PG-based Type I deicing fluids	Observations collected from personnel with experience in aircraft deicing operations: flight line personnel, deicing truck operator, flight crew, various DoD and contractor personnel. Flight crew visual inspection of surfaces for streaking and of windows (from inside) to ensure no degradation in visibility. Observation/Data sheets will be collected for each deicing event.	Post flight inspection shows surfaces to be substantially clear without large areas of ADF residue (esp. on the leading edge of the wings, in quiet areas and on windows).	Although fluid appeared to shear/flow from aircraft surfaces, post flight inspection indicated residue remained FAIL

Performance Objective	Data Requirements	Success Criteria	Results
Fluid exhibits slipperiness comparable to or less than that of PG on the deicing pad	Observations collected from field technician and government and contract observers on the flight line	No significant increase in slipperiness when walking or sliding shoes on pavement.	Fluid was observed to lead to significant slipperiness FAIL
Fluid has no impact on flight operations of the aircraft	Observations collected from flight crew	Flight control response, visibility, thrust(drag) and refueling boom operation are not compromised	Fluid did cause visual degradation on some windows during flight FAIL
Fluid requirements similar to PG	Observations from experienced deicing operator	Volume of fluid required for effective deicing is comparable or less than PG	Changing weather conditions prevented comparative evaluation with PG INCONCLUSIVE
Low slipperiness and visual degradation (wind tunnel test)	Measurement of fluid performance in wind tunnel testing	Slipperiness comparable to PG. Visual side-by-side measurements comparable to PG	No significant visual degradation, and slipperiness comparable to PG PASS
General/overall performance of fluid	Interviews of flight line operators, and flight crew	Performance suitable for recommendation to Base Commander	Demonstration participants concerned with residue issues

The first objective considers whether the deicing fluid can actually deice. The fluid should remove frozen contamination and should require the same, or less, effort, time and fluid quantity as the current standard process (utilizing a conventional PG fluid). This requires both objective and subjective evaluation. The process can be timed and fluid quantity can be measured, but unless the frozen contamination is uniform and consistent across aircraft and the environmental conditions are constant during the operation, these measurements may not provide a true comparison. Subjective evaluation by experienced maintenance personal will also be required. Success relative to this objective will be based on comparable process time and fluid use for EcoFlo II versus the conventional PG fluid and on observations by deicing operators.

Previous testing with alternative ADF formulations resulted in foaming of the fluid on the aircraft. This makes the visual determination of ice removal difficult and requires more time and attention on the part of the operator. The second objective requires the fluid to coat evenly and not foam. Evaluation of this objective will be based on the expertise of the deicing operator and the fluids ability to coat uniformly and not generate foam will indicate success.

Conventional PG Type I deicing fluids readily and cleanly leave aircraft surfaces soon after application. The fluid should not exhibit and viscous or dry residue. The third objective results from observations that some deicing fluids which reduce or eliminate PG do leave a residue. Post flight visual and tactile inspections will evaluate performance relative to this objective and success will require that no residue be detected.

The next two objectives, that slipperiness be comparable to current fluids and that the fluid not impact flight operations can be considered more specific symptoms of the previous objective. The fluid should leave no visible or invisible residue that increases slipperiness of aircraft surfaces. Maintenance personal will conduct post flight inspections to validate this objective. The fluid should also leave no residue that has an impact during flight or on flight performance of the aircraft. This includes blurring or windows or observation ports and contributing to any airflow or control issues during flight. Flight crew observations will measure this factor, with no discernible flight issues indicating success.

Table 4 Quantitative Performance Objectives

Performance Objective	Data Requirements	Success Criteria	Results
Fluid meets SAE AMS 1424G	Laboratory Testing per specification	Pass all 29 requirements under this specification	PASS
Fluid passes material compatibility testing	Test to the draft DoD Deicing Joint Test Protocol	Successful results in compatibility JTP tests	PASS
Measurable environmental benefits per deicing fluid decision support tool*	Facility characteristics, fluid use and runoff measurements – all input into Deicing Fluid Decision Support Tool	Positive environmental cost benefit results as indicated by Deicing Fluid Decision Support Tool	Decision Support Tool not utilized due to fluid failure in demonstration OBE

^{*} The deicing fluid Decision Support Tool is a MS Excel based tool developed under a previous effort. It features numerous fields for entry of ADF chemistry, usage and permitting data and provides a calculation of cost benefits of alternative ADFs versus conventional PG fluids.

The critical initial objective for a deicing fluid is that it meets the requirements specified in SAE AMS 1424G. This the governing document for commercial and military aircraft deicing fluids. Laboratory testing per the SAE specification will determine whether the fluid characteristic and performance requirements have been met.

Fluid compatibility with materials found on military aircraft but not typically found on commercial aircraft is not covered in SAE AMS 1424G, so a draft Deicing Joint Test Protocol has been created to cover many of those military unique materials. The second objective covers testing the fluid with these unique materials. Success is determined by comparison to conventional PG fluids in laboratory testing (i.e., no material property change or degradation greater than that exhibited by exposure to a conventional PG ADF).

The third objective is that the fluid has a measurable reduction in potential environmental impact when compared to a conventional PG fluid. This is effectively an environmentally driven cost benefits objective. The decision support tool prompts fluid property or chemistry, site permitting and site waste water handling data to be collected and provides calculations of potential cost savings resulting from implementation of alternative fluids. Success relative to the objective will be indicated by a notable potential cost savings.

4.0 SITE/PLATFORM DESCRIPTION

4.1 TEST PLATFORM/FACILITIES

Initially, the field demonstration was planned for Bangor, Maine, with the cooperation of the Maine Air National Guard (ANG). One request of that organization was that the demonstration involve an experienced flight test crew to safely perform the inflight portion of the evaluation. This necessitated a request for an Operational Assessment and led to the involvement of the Air Mobility Command Test and Evaluation Squadron (AMCTES) in the demonstration.

As coordination for the demonstration progressed, it became apparent that the Maine ANG would not be able to support the event (due primarily to an unusually short winter deicing season combined with operational commitments for aircraft). AMCTES was able to work with the 108th Wing, New Jersey Air National Guard at Joint Base McGuire-Dix-Lakehurst to undertake the demonstration.

Joint Base McGuire-Dix-Lakehurst was formed in Oct 2009 from McGuire AFB, Fort Dix and Naval Air Engineering Station Lakehurst, as a result of the 2005 Base Realignment and Closure (BRAC). The base encompasses 42,000 acres. In addition to housing the 108th Wing, McGuire field is home to the 305th Air Mobility Wing and the 514th Air Mobility Wing, and other mission partners.

The 108th Wing received its first KC-135 and began refueling missions in late 1991. The KC-135 Stratotanker has provided refueling and airlift for the US Air Force for over 50 years. The aircraft has a wingspan of over 130 ft. and a fuselage length just over 136 ft. It can carry 200,000 lbs. of fuel for transfer. The Air National Guard currently has 180 KC-135 aircraft in inventory.

4.2 PRESENT OPERATIONS

Many organizations and platforms operate out of Joint Base McGuire-Dix-Lakehurst. For the US Air Force, those occasionally requiring deicing services in addition to the 108th include the 305th Air Mobility Wing, operating KC-10s and C-17s. The base also hosts US Navy C-130s and C-9s, and civilian airlines that require deicing.

At Joint Base McGuire-Dix-Lakehurst, the last three deicing seasons (Oct 2009 to Feb 2012) have covered 4 to 6 months. During those combined seasons, the US Air Force issued 258,600 gal of Type I ADF concentrate (subsequently diluted to an approximate 50/50 mix with water for application).

The facility enlists Inland Technologies International, LTD for collection and recycling of fluids. Spent fluid, diluted with any water, slush or snow removed from the aircraft or present on the

flightline at the time of collection is collected and processed. During the last three deicing seasons, 105,531 gal of fluid mix was collected, and it's estimated that 15-20% of this is glycol.

4.3 SITE-RELATED PERMITS AND REGULATIONS

No permits were required specifically for the demonstration and no local regulations impacted the demonstration. EcoFlo II differs from current conventional PG deicing fluids only in the reduction of PG and the inclusion of non-hazardous alternatives. The reduction in PG (resulting in a reduced BOD and COD) and the elimination of hazardous additives results in a product with no additional regulatory and permitting burden. The result is anticipated to be similar wherever EcoFlo II might be implemented.

5.0 TEST DESIGN

The approach to this demonstration/validation involved three parts. The first two parts consisted of laboratory scale testing, and are discussed further in Section 5.1 below. The final part was the field demonstration on actual aircraft in winter deicing conditions, discussed in Section 5.2.

5.1 LABORATORY TESTING

Material compatibility testing was performed in order to ensure that EcoFlo is suitable for contact with unique military aerospace materials. Although SAE G-12 Aircraft Ground Deicing Committee has established commercial standards to which the fluid is tested, the DoD utilizes materials beyond those typical in the commercial world, and testing beyond the commercial standard must be considered. Material compatibility was evaluated per the Draft Deicing Joint Test Protocol by Concurrent Technologies Corporation.

Material compatibility testing was performed before the manufacturer made the decision to reformulate the ADF (i.e., laboratory testing was performed with EcoFlo rather than EcoFlo II); SMEs evaluated the two formulations and determined that the impact of the reformulation on material compatibility would likely be negligible. The material compatibility testing report is included as Appendix B.

The second part in the EcoFlo evaluation consisted of wind tunnel testing. The test was developed in response to concerns arising from previous formulations of this product, which left residue on the aircraft after deicing and flight. The on-aircraft demonstration of an ADF requires substantial planning and coordination, and it would have been advantageous to have foreseen any residue issues prior to undertaking those demonstrations. Unfortunately, there is no established laboratory or small scale procedure for measuring this performance factor.

A rough procedure utilizing panels attached to an automobile, in which ADF would be applied to the panels and the vehicle would be driven at high speed on an aircraft runway was initially considered. This approach was not promising due to the inability to approximate aircraft takeoff velocities and the difficulty in standardizing the procedure. The project team learned that Boeing would coincidentally be performing some evaluations of EcoFlo in a wind tunnel and took the opportunity to develop procedures to best evaluate fluid removal properties and residue impacts concurrently with that evaluation.

The investigation studied the condition of surfaces exposed to aircraft takeoff speed airflow in a wind tunnel subsequent to the application of EcoFlo or a conventional PG fluid. Transparent surfaces were evaluated for any impact on visual clarity attributable to ADF residue and painted aluminum surfaces were evaluated for slipperiness.

This evaluation was also performed before the manufacturer made the change from EcoFlo to EcoFlo II. As the reformulation to EcoFlo II was intended to improve the post application

properties of the fluid, it was assumed that any residue potential would only be decreased in the EcoFlo II. The wind tunnel test report is included in Appendix C.

5.2 TECHNOLOGY DEMONSTRATION

The third part of the demonstration/validation was the application of the fluid on actual aircraft during winter deicing conditions. The field demonstration was performed as an Operational Assessment (OA) by the Air Mobility Command Test and Evaluation Squadron (AMCTES), utilizing KC-135 aircraft at Joint Base McGuire-Dix-Lakehurst, NJ.

AMCTES developed the assessment protocol based on the OA Request and the demonstration plan, and with extensive coordination with the EcoFlo demonstration project team.

6.0 PERFORMANCE ASSESSMENT

6.1 LABORATORY TESTING

Material Compatibility Testing

The first part of the EcoFlo evaluation, testing the fluid compatibility with common military materials, was performed by Concurrent Technologies Corporation.

The EcoFlo fluid performed well, with the exception of percent volume swell of elastomeric materials (note that the conventional PG control fluid, Octaflo EF, also did not perform well with several of the elastomeric materials) and volume swell of low observable (LO) sealant, but with uncertainty over adequate cure of the sealant. Results are summarized in Table 4 below, with details in the CTC report in Appendix B.

Table 5 Material Compatibility Testing

Material Category	Test Method	Result
	Alternate Immersion	Pass
	Stress Corrosion Cracking	Pass
Metallic Materials	Total Immersion Corrosion	Pass
	Effect on Unpainted Surfaces	Pass
	In-plane Shear	Pass
	Barcol Hardness	Pass
PMC Material	Glass Transition Temp	Inconclusive
FIVIC Material	Sandwich Corrosion	Pass
	Thermal Oxidative Stability	Pass
	Percent Weight Gain	Pass
	UTS/Percent Elongation	Pass
	100% and 300% Modulus	Pass
Elastomeric Materials	Peel Strength/% Cohesive Failure	Pass
	Shore A Hardness	Pass
	Percent Volume Swell	Fail
Aircraft Wire Insulation	Immersion/Bend	Pass
Ancrait wire insulation	Voltage Withstand	Pass
Carbon-carbon Brake	Oxidation Resistance	Comparable to control
Infrared Windows	Change in transmission	Pass

LO Coatings	Liquid Uptake	Pass
	Adhesion	Pass (Some inclusive results – conspicuous failures for both control and EcoFlo)
	Pencil Hardness	Pass (Some inclusive results – conspicuous failures for both control and EcoFlo)
LO Sealant	Volume swell	Fail – potential cure issue
T 1 ' 1	Humidity	Pass
Lubricants and greases	Torque Rheometry	Pass
Cannon Plugs	Insulation Resistance	Unmated only – some failures
	Voltage Withstand Testing	Unmated only – some failures
Plastic Windows	Crazing Effect	Pass

The testing did result in a few inclusive results and failures. For polymer matrix composite (PMC) materials, the determination of glass transition temperature and how it is impacted by the ADF was inconclusive. CTC had difficulty locating samples of material in small quantities required for test (manufacturers would only provide large lots, at a cost beyond the budget of this project). Some material was located at Hill AFB, UT, but was not fully characterized. As the exact resin makeup was not identified, there was an inability to set the differential scanning calorimeter parameters accurately and the result was an indication of melting temperature, but not glass transition temperature.

For low observable (LO) coatings, there were also some inconclusive results in adhesion and hardness testing. In adhesion testing, one coating stack-up, the outer mold line primer with rain erosion topcoat, failed the cross-hatch adhesion test (ASTM D 3359, Method B) whether exposed or not exposed to EcoFlo (both passed the X-scribe adhesion test, ASTM D 3359, Method A). This indicates a possible discrepancy in test panel preparation rather than a failure attributable to EcoFlo.

For pencil hardness testing, measurements were performed before and after exposure to either the ADF or deionized (DI) water. For the coatings tested, hardness ranged from F at the soft end of the scale to H through 8H, with 8H as the hard end of the scale. The desired change in hardness is one unit or less (e.g., from 8H to 7H) after exposure. Two coating stack-ups, the outer mold line primer with anti-static rain erosion topcoat, and outer mold line primer with rain erosion topcoat averaged a change in hardness of 5 or 6 units. For both of these coating systems, the significant loss in hardness was measured both after exposure to either EcoFlo or to DI water, indicating potential issues with test panel preparation rather than a failure attributable to EcoFlo.

EcoFlo did appear to be absorbed by LO sealants as the volume of all samples increased, including one sample which increased by over 200%, after exposure to the ADF. If LO sealant compatibility is considered critical on a specific aircraft, these results would indicate the need for further evaluation prior to utilizing the fluid.

Similarly, testing indicated likely compatibility issues with electrical cannon plugs. On at least some of the evaluations for both the Insulation Resistance and Voltage Withstand testing results indicate that EcoFlo might either damage the insulation within the cannon plug, or leave some conductive contamination compromising insulated components.

The materials compatibility testing was not a formal pass/fail screening of the test ADF prior to a full scale demonstration. To be acceptable for use, even by military organizations, the critical qualification is compliance with SAE AMS 1424. The material compatibility JTP is significant as it covers evaluation of materials that may be present on military aircraft and are beyond those evaluated under the AMS document, but it does not convey or restrict authorization to use the fluid. For this project, failure of an ADF to demonstrate compatibility with some of the tested materials was considered more of an issue for attention and future detailed evaluation than a cause to preclude the demonstration.

Wind Tunnel Testing

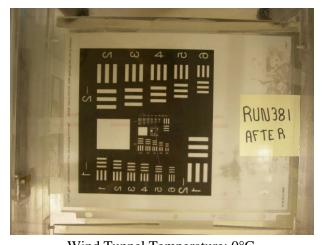
In the wind tunnel testing, surfaces (painted aluminum panels or the transparent bottom of the wind tunnel test section) were coated with either EcoFlo or a conventional PG fluid (Octaflo EF). Both fluids were initially diluted with water to form a 65% ADF/35% water (by volume) mixture. For some test runs, the fluid was then heated to reduce water content and approximate water loss due to spraying the heated fluid in actual operational use. The fluid was then applied in the controlled wind tunnel environment of either 0°C or minus -20°C. The wind tunnel was operated with an airflow of 65 m/s \pm 5 m/s in the test section (the wind velocity called out in SAE Aerospace Standard 5900 for the High Speed Ramp Test, based on takeoff conditions typical of large transport type jet aircraft) for a time previously established for the given set of test parameters.

To evaluate and document any degradation in clarity due to fluid residue on the test duct floor, after each run the test duct section was opened and a photograph was taken showing the eye chart through the Plexiglas floor. Visual clarity or resolution was evaluated for each set of test conditions in order to compare any degradation effects of the EcoFlo fluid with the PG fluid.

For slipperiness, a piston operated slip meter was utilized on the aluminum panels to determine contact angles at which a slip is likely to occur. The apparatus can be adjusted so that a polymer test foot is extended toward the surface at a controlled velocity and set angle. The test foot is mounted on a hinged fixture so it can slide along the surface after impact if a slip occurs, thus simulating a foot stepping onto a wet surface and possibly losing traction. The actual

measurement is determined by repeatedly adjusting the angle of impact until the test foot slips. As the measured angle approaches a normal to the surface, the surface is considered more slippery.

The results of wind tunnel testing showed that for all test conditions there was no notable impact on visual clarity for either the EcoFlo or the conventional PG ADF. A typical result is shown in Figure 2 below. As this surface was parallel to the airflow it is not a completely accurate approximation of the complex airflow around an aircraft, and it cannot guarantee that excess fluid residue will not gather on some aircraft windows or observation ports, but it does show that in this relatively simple approximation EcoFlo did not impact visual clarity any more than the PG ADF.



Wind Tunnel Temperature: 0°C H₂O Reduction Before Test; 0% Initial Fluid Thickness: 1 mm Wind Tunnel Run Time: 10 minutes

Figure 2 ADF Wind Tunnel Visual Clarity Test Configuration (EcoFlo ADF)

Measuring slipperiness was a difficult endeavor, as the interactions of surface profile, fluid properties, and dynamic factors of movement and impact are significantly complex. The measuring device itself affects the conditions at the point of impact, so it must be moved for each subsequent measurement, and during that time, evaporation and temperature changes may be influencing the fluid properties. It would likely require many repetitions of the test to attain statistically reliable and significant results, so the observations in this brief evaluation were, at best, approximations. Both fluids, however, did consistently leave significantly slippery surfaces after wind tunnel exposure. In some cases, but not all, the EcoFlo appears to be slightly more slippery then the conventional PG ADF, but in all cases, the surfaces were well beyond the threshold of what might be considered a safe walking surface (i.e., were unsafe surfaces).

The wind tunnel evaluation results suggest that EcoFlo is comparable to conventional PG ADF with respect to residue concerns. The project team understood that the evaluation limitations could not assuage all risk that the fluid would show performance discrepancies during the full, on-aircraft demonstration, but felt that this best effort at prescreening the fluid at least reduced that risk significantly, and that the full demonstration should proceed.

The wind tunnel report is included in Appendix C.

6.2 TECHNOLOGY DEMONSTRATION

The demonstration was accomplished through an Operational Assessment Request from Headquarters Air Force Materiel Command and was conducted by the Air Mobility Command Test and Evaluation Squadron with the support of the 108th Wing, New Jersey Air National Guard at Joint Base McGuire-Dix-Lakehurst.

The extensive coordination activities required for the demonstration pushed the schedule into the first few months of 2012, and an unusually mild winter was making deicing opportunities scarce. The EcoFlo demonstration team determined that AMCTES would perform the evaluation at the earliest opportunity, even if those team members not local to the base would not have time to travel and observe the event. If the opportunity presented itself, a second round of testing would be performed with more advance notification. AMCTES and the 108th Wing performed the demonstration on 9 February 2012.

AMCTES structured the evaluation with two assessment objectives, whether the fluid is potentially effective for use on a KC-135 aircraft and whether it is potentially suitable for use on a KC-135 aircraft. The first objective was judged by the time and quantity of fluid required to deice the aircraft, with a target being no greater time or fluid than needed for deicing with conventional PG fluids. The second objective was broken down into compatibility with the aircraft (whether the fluid flowed or sheared off surfaces and whether it obscured windows or viewing ports), compatibility with deicing equipment, and impact to safety as judged by test participants.

Weather conditions hindered an objective comparison between deicing effectiveness of the EcoFlo ADF and a conventional PG ADF. The skies transitioned from overcast to sunny prior to deicing the aircraft with PG fluid, allowing more radiant heating of aircraft surfaces and likely resulting in quicker removal of frozen contamination with less ADF. The performance relative to the criteria of equal or less time for deicing using equal or less fluid was rated as inconclusive, but deicing operators estimated that time and EcoFlo II required was consistent with their previous experience with that type of aircraft and frozen contamination. The EcoFlo II was rated as satisfactory for the first assessment objective, and thus potentially effective for use on KC-135 aircraft.

EcoFlo II ran into difficulties against the second assessment objective, suitability for use on the aircraft. The fluid was rated as satisfactory in appearing to shear/flow from aircraft surfaces and in maintenance test participants' rating of compatibility with aircraft surfaces, but unsatisfactory in aircraft window/viewing port visibility after ADF application. Also, although the fluid did appear to easily flow or shear from the aircraft, post flight inspection evinced a glossy appearance not seen on the conventional PG deiced aircraft.

The fluid was formulated to be compatible with all current deicing equipment, and the maintenance test team agreed by rating performance in that area as satisfactory.

With respect to safety concerns, maintenance test participants expressed concerns with the glossy, slippery residue remaining on the aircraft after flight. The increased slipperiness could be a potential fall hazard when performing servicing or aircraft inspections. For the safety of use criteria, the fluid performance was rated unsatisfactory and the EcoFlo II was determined to not be potentially suitable for use on a KC-135 aircraft.

Upon considering issues with window/viewing port visibility and safety concerns, AMCTES decided to perform no further demonstration activities with the current EcoFlo II formulation. The residue issues, something not new with non-PG or reduced PG ADF formulations, must be resolved before the Air Force would consider further evaluation of the fluid.

Full details of the demonstration are documented in the Final Report, KC-135 Compatibility with Low Biochemical Oxygen Demand Deicing Fluid, Operational Assessment. [2]

During post demonstration investigation efforts, one concern became the concentration of the ADF during the test. Prior to the demonstration, two totes (approximately 275 gal each) of EcoFlo II were added to the deicing truck and the fluid was diluted to 50% by volume. The refractometer available to the onsite test personnel was designed specifically for PG, so an accurate refractive index for the EcoFlo ADF could not be ascertained and concentration of the fluid could not be verified during the event. Subsequent to the demonstration, several samples of the diluted EcoFlo II were collected from the deicing truck and sent to the Aerospace Fuels Laboratory at Wright-Patterson AFB, OH. The first of these samples was found to have a refractive index equivalent to an approximately 80% solution of EcoFlo II in water. A second sample was collected; this time ensuring the fluid represented EcoFlo II from all levels, or depths, in the deicing truck tank, and was found to be closer to a 50% solution. Additionally, a sample was collected from the deicing truck nozzle and found to have a refractive index equivalent to a nearly 50% solution.

This was not a statistically significant sample size, and it is unclear whether the first measurement was an outlier or truly representative of the tank contents (EcoFlo II is formulated to be fully miscible in water and settling of ADF components should not have been a factor). Additionally, the earliest samples were collected 28 February 2012, more than two weeks after

the demonstration, so a direct representation of the fluid conditions at the time of the demonstration cannot be assumed. Further analysis to characterize any unanticipated fluid behavior that may have impacted the demonstration has not been performed.

7.0 COST ASSESSMENT

With the EcoFlo II ADF not performing in critical areas of the evaluation and the Operational Assessment cut short, the collection of comprehensive data to support a detailed analysis of environmental cost factors was not completed. For a rough cost comparison, qualitative factors such as the cost of EcoFlo II or waste water handling at Joint Base McGuire-Dix-Lakehurst might be considered.

EcoFlo II is formulated to be a drop in replacement for conventional PG ADFs. There should be no changes required for equipment or operational procedures. During the demonstration, a one-to-one comparison between EcoFlo II and PG was inconclusive, due to rapidly changing weather (i.e., the frozen contamination was not as heavy during the PG operation), but operators did not notice any significant or conspicuous ineffectiveness in deicing when applying the EcoFlo II. This would indicate that in general, implementation costs would be negligible.

For raw material costs, with both EcoFlo II and PG containing a significant quantity of PG, the fluids could be anticipated to remain similar. Additionally, the manufacturer has indicated an intention to price EcoFlo II similarly to their conventional PG fluids.

Environmentally related costs may be more complicated to determine. EcoFlo could be expected to lower permitting costs and liability risks, as the BOD and COD are less than that of PG. At Joint Base McGuire-Dix-Lakehurst, however, waste PG is currently collected with a vacuum truck and recycled. Factors such as the market demand for recycled PG and the ability to recycle EcoFlo II would need to be considered in calculating the cost comparison between handling PG waste and handling EcoFlo II waste. Conceivably, a high demand for PG combined with any difficulty in recycling EcoFlo II could result in higher costs when handling the EcoFlo II waste.

An additional cost factor to consider would be the investment in demonstrating an alternative ADF. As this project illustrates, attempts to investigate fluid and residue behavior on aircraft surfaces (i.e., visibility degradation and slipperiness), are still unreliable. The pre-demonstration wind tunnel testing did not reveal a high risk for residue issues and indicated that EcoFlo could be anticipated to perform similarly to PG. Through the KC-135 demonstration residue was found to still be a problem. Development of a more reliable, laboratory scale methodology for predicting alternate ADF behavior might help reduce the investment cost by adding certainty prior to coordinating and executing a full field demonstration with pre and post flight evaluations.

7.1 COST MODEL

The project team anticipated use of a deicing fluid Decision Support Tool, developed under a previous ADF demonstration, to analyze costs factors and determine the potential cost benefits resulting from implementation of EcoFlo II. A sample of some of the data to be collected in the tool is included in Figure 3. As discussed in the Section 7.2, ultimately this tool was not used.

EVALUATION WORKSHEET FOR ALTERNATIVE AIRCRAFT DEICERS	
This evaluation tool was designed to assist a Base Environmental Manager (EM) in assessin compliance, and cost implications of a new Type I ADF formulation that is being considered a in use. The evaluation is at a screening level, intended to give the EM a sound indication of the changes and benefits that can be expected with a switch to the alternative ADF. This informal regarding a switch to the new formulation.	as an alternative to the Type I ADF currentl he general direction and magnitude of
It is essential to understand that the tool is not intended to replace more sophisticated analys demonstrations of regulatory compliance or engineering design of deicing runoff manageme	
SITE INFORMATION	
Site Name	
Pittsburgh ANG	
Address	
Person filling out form	
Chris Cieciek, Limno Tech on behalf of LTC John Towers	
E-mail Address	Telephone Number
ccieciek@limno.com	
CURRENT SITUATION	
NPDES Storm Water Permit Information	
1 Does your site have an NPDES Storm Water permit for discharge of deicing runoff?	1 TYES C NO
2 NPDES permit number	2
3 Permitting authority	3
Permit limits during periods of peak deicing activity	BOD5 COD
4 Most stringent permitted discharge concentration (mg/L). Leave blank if there are no limit	its. 4
5 Most stringent permitted maximum daily load (lbs/day). Leave blank if there are no limits.	5
Current Type I Deicer Information (See MSDS and manufacturer's literature)	Type I
6 Decay rate at 20°C (1/day)	6 0.18
7 BOD5 concentration of propylene glycol (mg/L)	7 650,000
8 Percent glycol in purchased product	8 88.00%
9 BOD5 concentration in the purchased product (mg/L)	9 572,000
10 96-hour aquatic toxicity (LC50) for fathead minnows (mg/L)	10 10,800
11 48-hour aquatic toxicity (LC50) for daphnia (mg/L)	11 14,000
12 Aquatic toxicity (LC50) for other organisms (mg/L)	12
13 Name of other test organism	13 Teet

Figure 3 Decision Support Tool Example

7.2 COST ANALYSIS AND COMPARISON

Due to the scarcity of icing weather and resulting short notice for the initial demonstration, non-local team members were not able to travel to Joint Base McGuire-Dix-Lakehurst and collect data to utilize the tool. Effectively, once it was determined that the fluid raised some safety concerns, and the demonstration would not be repeated, the team acknowledged that there would no longer be significant value in attempting to visit Joint Base McGuire-Dix-Lakehurst to attempt to collect or estimate this data.

8.0 IMPLEMENTATION ISSUES

As an SAE AMS 1424 Type I certified deicing fluid, compatible with current deicing equipment, EcoFlo II is designed to be a drop in replacement for Conventional PG ADFs, and should have no significant implementation issues.

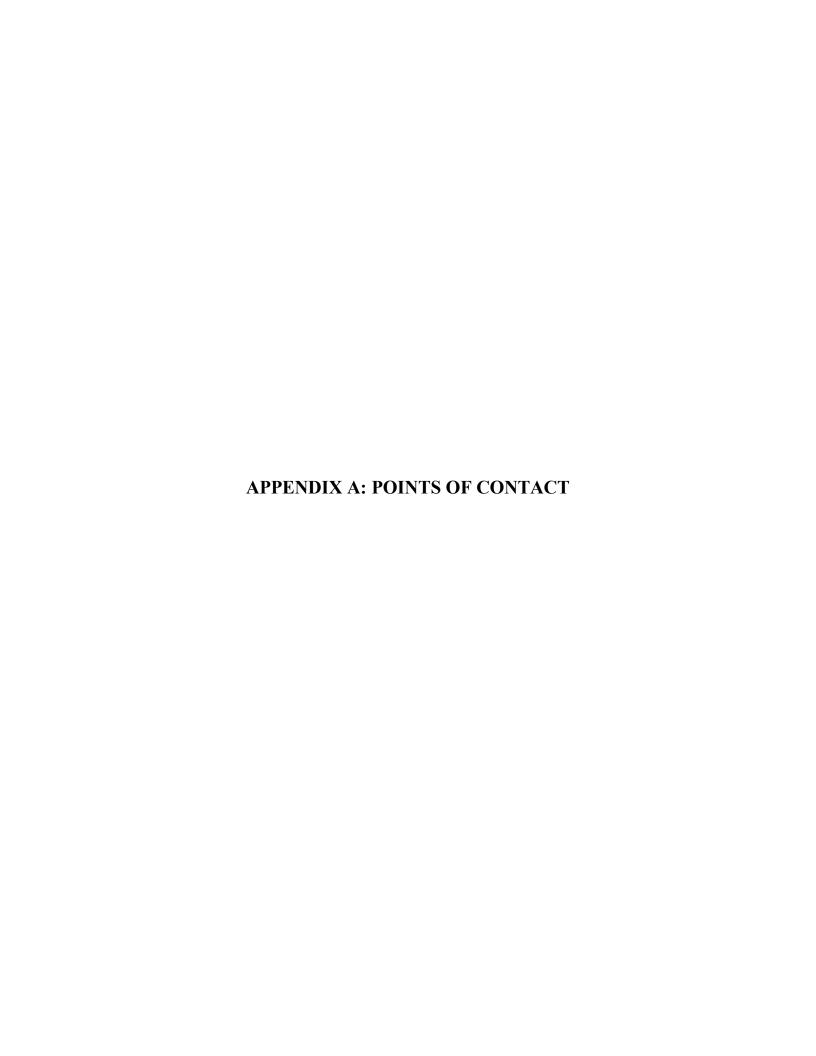
The evaluation of compatibility with military materials did indicate a few areas of concern, and it would be recommended that those undergo further evaluation prior to application to aircraft utilizing those materials. Risk of exposure and degree of potential damage or degradation to the material should be analyzed and understood.

The primer impediment to implementation is the apparent residue which can obscure window/viewing ports and leave aircraft surfaces excessively slippery, causing safety concerns during post flight inspection and maintenance. This is not a new concern, and in this project it prompted the incorporation of a wind tunnel test to hopefully identify that characteristic prior to the full scale demonstration. Considerations of alternative ADFs for future implementation should research mechanisms causing this undesired performance trait and effective small scale or laboratory procedures to ensure it has been controlled or eliminated, prior to expending the time and cost on a full demonstration.

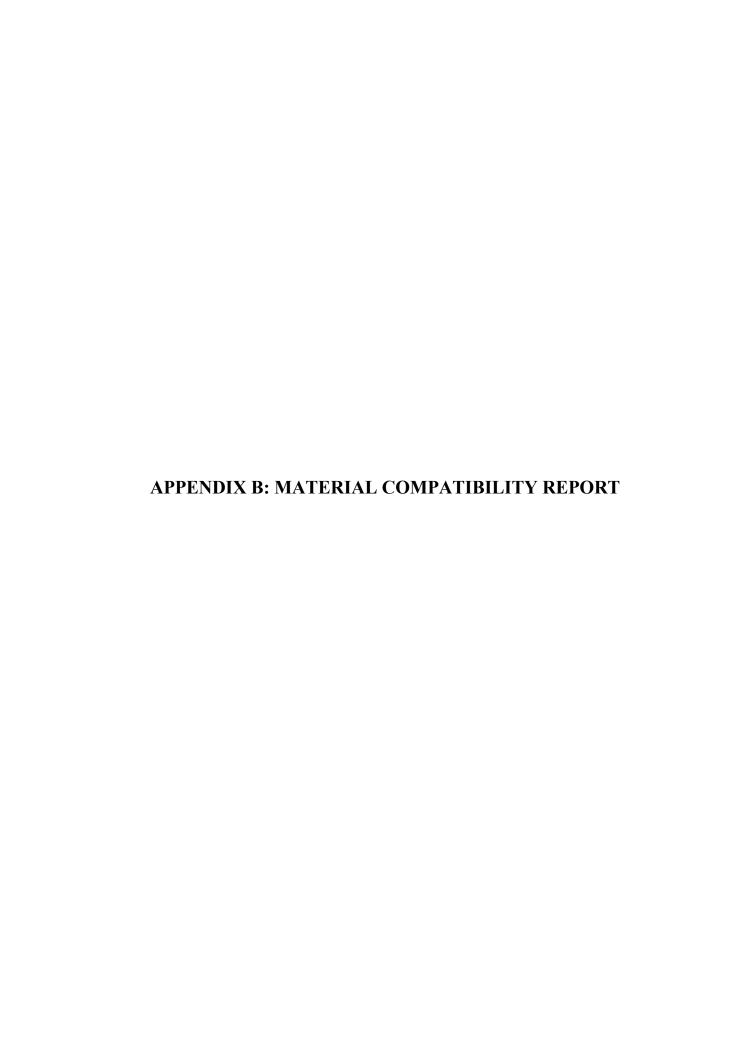
If an EcoFlo related ADF formulation is eventually found suitable for use on military aircraft a minor issue to consider might be integrating the new materials into the waste fluid handling process, especially at a facility like Joint Base McGuire-Dix-Lakehurst where PG is currently collected and recycled and a reduced PG fluid might be of less value to the recycling vendor.

9.0 REFERENCES

- [1] SAE International. Aerospace Material Specification AMS 1424G, Deicing/Anti-Icing Fluid, Aircraft, SAE Type I. 18 January 2006 (Available from SAE International, website: standards.sae.org/ams1424g)
- [2] AMC Test and Evaluation Squadron. AMC Test 10-001-12, KC-135 Compatibility with Low Biochemical Oxygen Demand Deicing Fluid, Operational Assessment, Final Report. June 2012.
 - (Available from HQ AMC/TE, 402 Scott Drive, Unit 1A5, Scott AFB IL 62225-5364)



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Eco Flo Deicer Material Compatibility Test Report

June 15, 2011

Submitted by

CTC Concurrent Technologies Corporation

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APPENDIX

A: Torque Rheometry Report from ATS Rheosystems

LIST OF ACRONYMS

Al Aluminum

ALON Aluminum Oxynitride

AMS Aerospace Material Standard

BMI Bismaleimide

BOD Biochemical Oxygen Demand

C Celsius Co Cobalt

COD Chemical Oxygen Demand

CTC Concurrent Technologies Corporation

DI Deionized

DSC Differential Scanning Calorimetry EDM Electrostatic Discharge Machining

F Fahrenheit

FTIR Fourier Transform Infrared Spectrometry

HA Hardness HIPOT High potential

HVOF High Velocity Oxygen Fuel

IR Infrared

JTP Joint Test Protocol LO Low Observable MEK Methyl Ethyl Ketone

mg milligrams
MPa MegaPascals
PAO Polyalphaolefin

PFPAE Perfluoropolyalkylether

PG Propylene glycol

PMC Polymer Matrix Composite
rpm revolutions per minute
RSD Relative Standard Deviation
SAE Society of Automotive Engineers

SAIC Science Applications International Corporation

SCC Stress Corrosion Cracking

SCCM Standard Cubic Centimeter per Minute

V volts, Vanadium V/s volts/second

VAC Volts Alternating Current

WC Tungsten carbide

1.0 INTRODUCTION

Science Applications International Corporation (SAIC) contracted Concurrent Technologies Corporation (*CTC*) to perform materials compatibility testing, as listed in the Joint Test Protocol (JTP) for Aircraft/Runway Deicers. This JTP is a compilation of substrate materials and testing methods that extend beyond the current Society of Automotive Engineers (SAE) specifications that are used to qualify deicing materials for commercial use. These substrate materials are, in many cases, unique to military aircraft, and the testing of compatibility of these substrates with the candidate deicing fluid is required by the equipment single managers.

This report lists the substrate materials analyzed, testing methods used, and results of the materials compatibility evaluations for the EcoFlo Type I aircraft deicer. Any deviations to the testing methods are noted, and the EcoFlo test results are compared to previous test results, if available, for standard Type I propylene glycol (PG)-based deicer, Octaflo EF. In some instances, data with Type I PG deicer was generated while conducting this testing, if testing or substrate materials differed from previous testing events.

2.0 CANDIDATE DEICER – ECOFLO

EcoFlo, manufactured by Octagon Process, Inc., is a Type I de-icing fluid qualified to SAE AMS 1424. EcoFlo is a hybrid deicing fluid that reduces propylene glycol by substituting glycerin in proprietary concentrations. According to the manufacturer, EcoFlo is the only deicing fluid that reduces the chemical oxygen demand (COD) by 25% and the biochemical oxygen demand (BOD) by 35% compared to traditional propylene glycol based deicing fluids. Table 1 contains the physical and chemical properties of this fluid.

Table 1. Physical and Chemical Properties of EcoFlo

Physical/Chemical Property (Test Name)	Test Result
Freezing Point (concentrate)	-26°C
Surface Tension	37-47 dynes/cm
Lowest Operational Use Temperature (65:35 dilution)	-30.5°C
Flash Point	>100°C
Specific Gravity at 25°C	1.14
Refractive Index	1.4375-1.4405
рН	7.2-8.2
Chemical Oxygen Demand (COD)	1.20 kg O ₂ /kg fluid
Biochemical Oxygen Demand (BOD)	0.39 kg O ₂ /kg fluid at 20°C

The Octaflo EF project, used as the standard Type I baseline deicer, is also manufactured by Octagon Process, Inc.

3.0 TESTING OVERVIEW

This section lists the substrate materials and testing methods that were performed as part of this effort. Table 2 lists the substrate materials for each test category.

Table 2. Substrate Materials Tested per Category

Test Category	Substrate Material
	4140 Steel (AMS 6395)
	9Ni-4 Co steel (AMS 6523)
Metallic Materials	7075-T6 Bare Aluminum Alloy (AMS 4045H)
	AZ91E-T6 Cast Magnesium Alloy (AMS 4446A)
	C63200 Aluminum-Nickel-Bronze (AMS 4640)
Polymer Matrix Composites	Supplied by depot (SAIC-coordinated)
	Nitrile Seal Material (MIL-R-6855, Class I)
	Neoprene Seal Material (MIL-R-6855, Class II)
Elastomeric Materials	Polysulfide Sealant (MIL-S-8802, Type I)
	Corrosion-Inhibiting Sealant (MIL-PRF-81733D)
	Polythioether Sealant (AMS-3277)
	High Temp Polysulfide Sealant (AMS-3276C)
	Polyimide (MIL-W-81381/11-20)
A in a sec Co XVI no. To a section in	Teflon (MIL-W-22759/11-20)
Aircraft Wire Insulation	Hybrid Construction (MIL-W-22759/86-20)
	Cable-insulated twisted pair (MIL-W-22759)
	CARBENIX 1000 with and without antioxidant coating
Carbon-carbon Brake Friction	CARBENIX 2000 with and without antioxidant coating
Materials	CARBENIX 2330 with and without antioxidant coating
	CARBENIX 4000 with and without antioxidant coating
Infrared (IR) Window	Aluminum oxynitride (ALON)
Materials	Sapphire – uncoated
	MS-133 Outer mold line primer (PRC Desoto)
Low Observable (LO)	MS-424 Inner mold line primer (Deft)
Coatings	MS-484 Anti-Static Rain Erosion Urethane (CAAP CO)
	MS-485 Rain Erosion Urethane (CAAP CO)
LO Sealant	PR 2200, Class B, gap sealant
	MIL-PRF-32014 Polyalphaolefin ((PAO) based grease)
	MIL-PRF-81322 (PAO based grease)
	MIL-PRF-27617 Perfluoropolyalkylether ((PFPAE)
	based grease)
Lubricants and greases	MIL-PRF-83261 (silicone oil based grease)
	MIL-PRF-87257 lubricant
	MIL-PRF-83282 lubricant
	MIL-PRF-5606 lubricant
	MIL-PRF-7808 lubricant

Cannon Electrical Plug Pins	MIL-STL-38999 Series III subminiature cylindrical type
Calmon Electrical Flug Fills	connectors
HVOF Coating	83% WC-17% Co
Plastic Windows	MIL-P-5425 (cast acrylic sheet, heat resistant)

Table 3 lists the testing procedures that were used to evaluate each material category. Each of the testing procedures is described in detail, in the next section.

Table 3. Testing Procedures per Category

Test Category	Testing Procedure
	Alternate Immersion (ASTM G-31)
Metallic Materials	Stress Corrosion Cracking (ASTM G-44)
Metanic Materials	Total Immersion Corrosion (ASTM F483)
	Effects of Unpainted Surfaces (ASTM F485)
	Density & Specific Gravity (ASTM D792)
	Fiber Content (ASTM D3171)
	In-plane Shear (ASTMs D3518 & 3039)
Polymer Matrix	Barcol Indentation (ASTM D2583)
Composites	Glass Transition Temperature (ASTM E794)
	Sandwich Corrosion (ASTM F1110)
	Thermal Oxidative Stability (Draft JTP)
	Percent Weight Gain After Soak (Draft JTP)
	Ultimate Tensile Strength (SAE 5127/1)
	Percent Elongation (SAE 5127/1)
Elastomeric Materials	100% and 300% Modulus (SAE 5127/1)
Elastometic Waterials	Peel Strength and % Cohesive Failure (SAE 5127/1)
	Shore A Hardness (ASTM D2240)
	Percent Volume Swell (Draft JTP)
	Conductivity (Draft JTP, EPA 120.1)
Aircraft Wire Insulation	Immersion – Swell (SAE 4373, TM 601)
Affectant whe insulation	Bend Test (SAE 4373, TM 714)
	Voltage Withstand Test (SAE 4374, ASTM 3032)
Carbon-Carbon Brake	Cyclic Heating Procedure (Draft JTP)
Friction Materials	Shore D Hardness (ASTM D2240)
Infrared Window Materials	Change in Infrared Transmission (Draft JTP)
LO Coatings	Pencil Hardness (ASTM D3363)
	Tape Adhesion (ASTM D3359, "A" and "B")
	Liquid Uptake (ASTM D570)
LO Sealant	Volume Swell (SAE 5127/1)
Lubricants and Greases	Humidity Test Procedure
	Torque Rheometry (Draft JTP)
Cannon Electrical Plug	Insulation Resistance (MIL-STD-1344A, 3003.1)
Pins	Shell-to-shell Conductivity (MIL-STD-1344A, 3007)

	Dielectric Withstanding Voltage (MIL-STD-1344A, 3001.1)
HVOF Coating	Alternate Immersion (ASTM G44-99)
	Humidity Testing (ASTM D1748-02)
Plastic Windows	Crazing Effect (ASTM D484)

4.0 TESTING METHODS AND RESULTS

Each of the following sections describes the substrate materials that were involved in the material compatibility testing and the test methods that were used to evaluate the effects of the deicing materials on the substrates. The test results are then provided, as well as any deviations to the described testing methods.

4.1 **Metallic Materials**

The purpose of testing the metallic materials was to determine the effect of the EcoFlo deicer on the metallic components of aircraft or ground support equipment that will be exposed to the deicing solution. The testing methods deal with cyclic exposure to deicing solution in order to simulate deicing conditions.

4.1.1 Metallic Substrate Materials

Metallic substrates were chosen that comprise components of aircraft and/or ground vehicle equipment that could come into contact with the deicing chemical. The specimens included 2.5 x 2.5-inch and 1 x 2-inch flat test panels for immersion testing, 2 x 6-inch flat test panels for effect on unpainted surfaces, and tensile test bars for stress corrosion cracking. The substrates and specification numbers are listed in Table 4.

Substrate **Specification Number**

AMS 4911

Table 4. Metallic Materials

4140 Steel AMS 6395 7075-T6 Bare Aluminum Alloy AMS 4045H AZ91E-T6 Cast Magnesium Alloy AMS 4446A C99300 Aluminum-Bronze Alloy AMS 4640 (alloy is closest representative)

4.1.2 Metallic Materials Testing Methods

Titanium 6Al-4V

Four test methods were used to assess the effects of the deicing solutions on metallic materials:

- 1. Alternate Immersion Testing ASTM G-31
- 2. Stress Corrosion Cracking ASTMs G-44 and G-49
- 3. Total Immersion Corrosion ASTM F483 and ADS-61A-PRF
- 4. Effects on Unpainted Surfaces ASTM F485 and ADS-61A-PRF

4.1.2.1 Alternate Immersion

Test Description

Three specimens of each test metal were machined into 5 x 5-centimeter squares using electrostatic discharge machining (EDM) wire to avoid localized heating. The surface of the specimens was ground to a 32 micro-inch (µin) finish, wiped clean with methyl ethyl ketone (MEK) and weighed to the nearest milligram (mg) on an analytical balance. The specimens from a single test metal were placed in the stress corrosion cracking chamber, to avoid potential cross contamination of corrosion product(s). The chamber was programmed with the following parameters: 1) submerge specimens in the deicing fluid for 10 minutes, 2) drain and then air dry specimens for 50 minutes, and 3) repeat steps one and two continuously for three weeks. The specimens were checked for corrosion daily, and once corrosion was detected, the specimens were removed from the chamber. After removal from the chamber or at the end of the test run, the specimens were weighed and examined for staining, pitting, exfoliation, and corrosion product buildup. All specimens were digitally photographed.

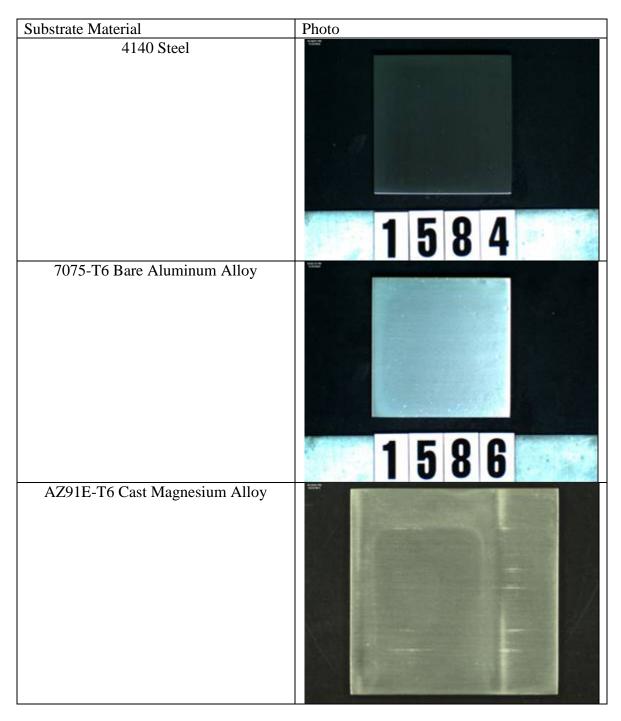
Test Methodology

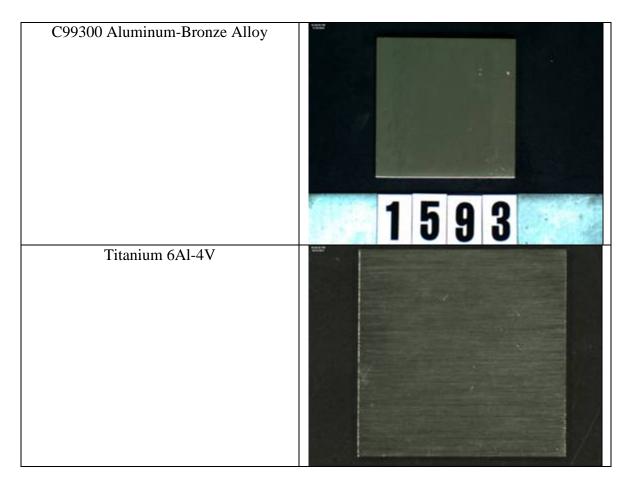
Parameters	5 x 5-centimeter specimens with a three-week cycle
	of 10 minutes submerged in the deicing solution and
	50 minutes air dry
Type/Number of Specimens	3 specimens for each substrate material per deicing
	solution as listed in Table 4
Experimental Control Specimens	None
Acceptance Criteria	No corrosion present or staining, pitting, exfoliation
	due to corrosion.
Reference Document	ASTM G-31
Test Equipment	Stress Corrosion Cracking Chamber
	Analytical Balance

Test Results

Table 5 contains representative photos of the alternate immersion specimens. There was no noticeable change in weight or corrosion/oxidation of the metallic substrates as a result of exposure to the Ecoflo deicer.

Table 5. Alternate Immersion Photos – After Exposure





Test results from Type I Octaflo deicer from a previous testing effort was reviewed and compared to this data. The PG deicer also had little to no effect on these metallic substrate materials.

4.1.2.2 Stress Corrosion Cracking

Test Description

Stress corrosion cracking (SCC) tests was performed in accordance with ASTM G-44 and the additional procedures outlined in the JTP. Samples were prepared according to ASTM G-49, Standard Practice for Preparation and Use of Direct Tension Stress-Corrosion Test Specimens. The samples were placed into frames and loaded into a special stressing fixture. An extensometer was used to determine the strain applied to the specimen (target is near 80 % of the measured yield strength). To ensure there are no galvanic effects between the frame and the specimen, the frame and threaded ends of the specimen were coated with stop-off lacquer or beeswax. The only metallic material exposed to the anti-icing solution was the test specimen.

Once the framed samples have air dried, they were placed in the SCC chamber with the alternate immersion samples of the same alloy, when possible. The same cycle time applies to the SCC samples: 1) submerge for 10 minutes in deicing solution, 2) drain and air dry for 50 minutes, and 3) repeat steps one and two continuously for three weeks. The samples were removed from the

chamber before the end of the test time if catastrophic failure occurred (the specimen fractures). Otherwise, visual examinations were conducted to determine if any cracking, pitting, or other discoloration occurred due to exposure.

Test Methodology

Parameters	SCC tensile specimens were loaded to approximately 80% strain and tested in a 3-week cycle of 10 minutes submerged in the deicing solution and 50 minutes air dry	
Type/Number of Specimens	3 specimens for each substrate material as listed in Table 4	
Experimental Control	None	
Specimens		
Acceptance Criteria	No fracture while the specimen was strained or during	
	removal from the frame at the end of testing.	
Reference Document	ASTM G-44, ASTM G-49	
Test Equipment	Stress Corrosion Cracking Chamber	
	Tensile Test Machine	
	Extensometer – 1 inch	
	Extensometer Calibrator	
	Mechanical Vise	

Test Results

The average pass/fail results for each metal are listed in Table 6.

Table 6. Stress Corrosion Cracking Results

Substrate	Test Result	Observations
4140 Steel	PASS	No cracking, pitting, discoloration, or fracture
7075-T6 Aluminum (Bare)	PASS	No cracking, pitting, discoloration, or fracture
AZ91E-T6 Cast Magnesium Alloy	PASS	No cracking, pitting, discoloration, or fracture
C99300 Aluminum-Bronze Alloy	PASS	No cracking, pitting, discoloration, or fracture
Titanium 6Al-4V	PASS	No cracking, pitting, discoloration, or fracture

As noted, all materials passed SCC testing, with no failures of the specimens and no visible changes to specimen appearance. In addition, the results are the same as the SCC results for the Octaflo Type I deicer.

4.1.2.3 Total Immersion Corrosion

Total immersion corrosion testing was conducted on panels measuring 2 x 1 x 0.06 inches, with a 0.125-inch diameter mounting hole located at one end of the panel. Four specimens of each

substrate material were required for testing with each deicing fluid. The test panels were precleaned by immersing them in Type II mineral spirits, followed by several dip immersions into MEK. The excess solvent was drained from the test panels and the panels were then placed in an oven at 120 ± 5 °C for 15 minutes to dry.

Following pre-cleaning, three of the four specimens of the same alloy were weighed to the nearest 0.1 mg. The three weighed specimens of each alloy were then immersed in the deicing fluid at $38 \pm 3^{\circ}$ C ($100 \pm 5^{\circ}$ F) for 24 hours. Only specimens of the same alloy were placed together in a vessel. The fourth specimen of each alloy was retained for comparison purposes. At the end of the 24 hours, the test specimens were removed from the solution and rinsed thoroughly under hot tap water at 49 to 60° C ($120 \text{ to } 140^{\circ}$ F). The panels were then rinsed in room temperature deionized (DI) water (DI water conforming to ASTM D-1193, Type IV specification is required). Following the DI water rinse, the test panels were then rinsed with a stream of acetone from a wash bottle and oven dried at 120° C (250° F). The test panels were removed from the oven, desiccated until cooled to ambient, and then weighed once again. Digital images of the panels were captured to document the surface condition. The panels were also examined for the following visible changes in comparison with the fourth untreated specimen of each alloy, and the results were recorded:

- Discoloration and dulling,
- Etching,
- Presence of accretions and relative amounts,
- Pitting, and
- Presence of selective or localized attack.

The panels were immersed in the same test solution for an additional 144 hours, rinsed, and weighed. The visual examination was again performed, with digital photographs taken as needed. Weight loss/gain and visual observations were recorded at each time interval.

Test Methodology

Parameters	2 x 1 x 0.06 inch panels weighed, immersed in deicing
	fluid for 24 hours and then an additional 144 hours.
Type/Number of Specimens	4 panels of each metallic material in Table 4
Experimental Control Specimens	Test panel not exposed to the deicing fluid
Acceptance Criteria	Minimal visible changes; weight change ≤ 0.5%
Reference Document	ASTM F483, ADS-61A-PRF
Test Equipment	Mechanical Convection Oven
	Thermometer
	Constant Temperature Bath

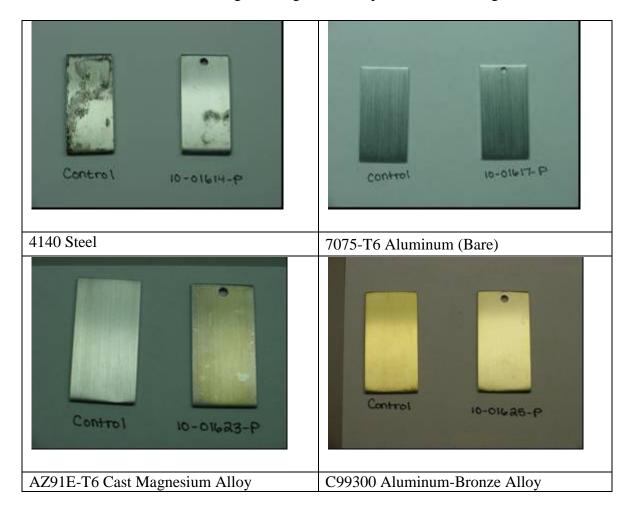
Test Results

The average weight changes for 24 and 168-hr for each substrate is listed in Table 7. Figure 1 contains representative photos.

Table 7. Total Immersion Corrosion Results

Substrate	Average Weight Change (g) After 24 Hours	Standard Deviation	Average Weight Change (g) After 168 Hours	Standard Deviation
4140 Steel	0.00003	0.0001	-0.00007	0.0005
7075-T6 Aluminum (Bare)	0.00003	0.0003	-0.0003	0.0001
AZ91E-T6 Cast Magnesium Alloy	-0.0001	0.0002	-0.00007	0.0003
C99300 Aluminum- Bronze Alloy	0.0000	0.0000	-0.00057	0.0005
Titanium 6Al-4V	-0.0003	0.0003	-0.0006	0.0003

The EcoFlo deicer appeared to have minimal effect on the various metallic materials. All materials exhibited less than 0.5% change in weight when exposed to the deicing solution.



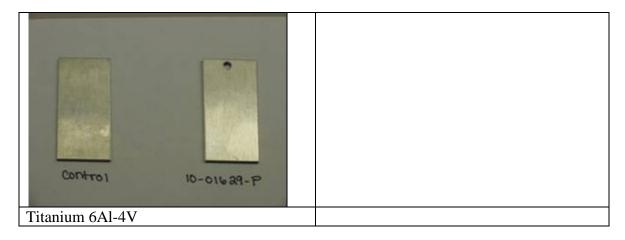


Figure 1. Photos of Metallic Materials after 168-hour Immersion

4.1.2.4 Effects on Unpainted Surfaces

Test Description

To determine the effect of deicing solutions on unpainted specimens, test panels measuring 2 x 6 x 0.020 inches were prepared by cleaning with MEK. Two test panels of each alloy were exposed to EcoFlo deicer. The test panels were immersed for three to five minutes in a sufficient quantity of deicing solution to cover approximately one half of the panel. After removing the panels from the test solution, the panels were immediately placed at an angle of 45° from the horizontal in a mechanical convection oven that is maintained at $65.5 \pm 2^{\circ}$ C ($150 \pm 5^{\circ}$ F). After 30 minutes, the test panels were removed and cooled to room temperature. Within 15 minutes after cooling, each panel was rinsed on each side under running tap water for one minute without using mechanical agitation, followed by rinsing on each side with distilled or DI water from a squeeze bottle for 15 seconds. The panels were air dried for 30 minutes and visually examined for etching, staining, or the presence of residue. Digital photographs were taken to document the results.

<u>Test Methodology</u>

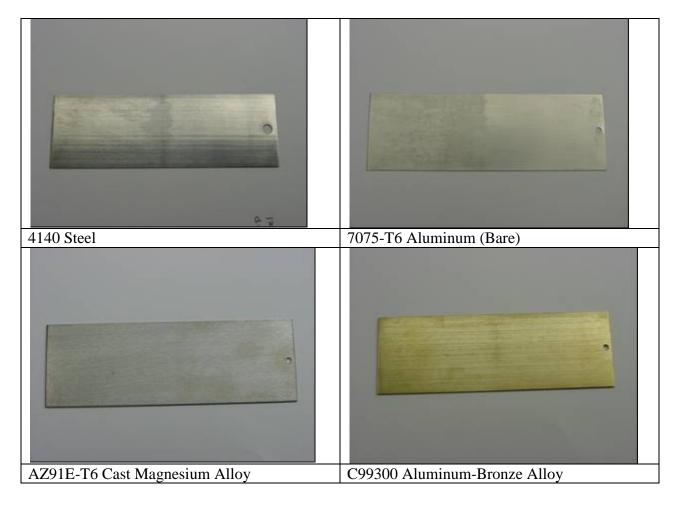
Parameters	2 x 6 x 0.02 inch panels immersed in deicing fluid for	
	3-5 minutes, dried at 150°F, cooled, rinsed with DI	
	water, air dried, and visually inspected	
Type/Number of Specimens 2 panels of each metallic material listed in Table		
Experimental Control Specimens Portion of the test specimens not exposed to dei		
	fluid	
Acceptance Criteria	Minimal visible changes	
Reference Document ASTM F485, ADS-61A-PRF		
Test Equipment	Mechanical Convection Oven	

Test Results

The average pass/fail results for each metallic material are listed in Table 8. These results are visual interpretation of the effects of exposure, followed by intense heat, on the substrate surface. Representative photos are located in Figure 2.

Table 8. Results for Effects on Unpainted Surfaces

Substrate	Test Result	
4140 Steel	PASS – slight staining	
7075-T6 Aluminum (Bare)	PASS – slight staining	
AZ91E-T6 Cast Magnesium Alloy	PASS	
C99300 Aluminum-Bronze Alloy	PASS – slight staining	
Titanium 6Al-4V	PASS – slight staining	



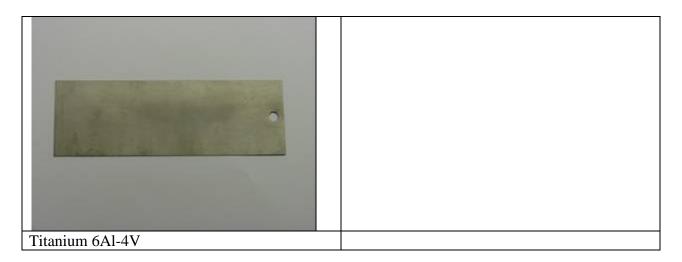


Figure 2. Photos of Effects of Painted Surfaces Panels after Test

All substrates received a PASS rating when exposed to the deicing solution indicating no effects of the deicer on the metallic material. From the photos in Figure 2, minimal staining can be noted on the lower portions of the panels, which was the area exposed to the deicer. However, nothing beyond staining was noted for this exposure, so results are considered passing.

Overall, the results of testing metallic materials under various exposure scenarios with EcoFlo deicer showed minimal effect of the deicer on the metallic substrates. These test results were consistent with previous results from testing Octaflo Type I PG-based deicer.

4.2 Polymer Matrix Composites

Polymer matrix composites (PMCs) are systems of polymer resins with a reinforcing material, such as glass or carbon. PMC incorporates the forming and protective properties of resins and the high tensile strength properties of reinforcing materials to create a high strength, moldable, environmentally resistant, low-density material that is used as a replacement for metals.

4.2.1 Polymer Matrix Composite Substrate Materials

One PMC material was tested under this effort. The PMC material was provided by Hill Air Force Base, through the coordination of SAIC, to *CTC*. The sample provided was a sheet of used material, 3/16-inch in thickness. Samples were cut from this sheet in *CTC*'s machine shop, with all cutting fluid promptly removed with acetone.

4.2.2 Polymer Matrix Composite Test Methods

Material compatibility testing of the PMCs was evaluated using the following methods:

- 1. Density ASTM D 792 (tag end test)
- 2. Fiber Content ASTM D 3171 (tag end test)
- 3. In-plane Shear ASTM D 3518 and 3518M (mechanical test)
- 4. Glass Transition Temperature ASTM E794 (tag end test and physical test)

- 5. Barcol Indentation ASTM D 2583 (physical test)
- 6. Sandwich Corrosion test ASTM F 1110 (physical test)
- 7. Thermal Oxidative Stability JTP (physical test)
- 8. Percent Weight Gain after Soak JTP (physical test)

As noted, the testing included tag end testing of the initial composite sheet quality and mechanical and physical testing. Testing of the polymer matrix composite sheet was conducted before deicer exposure to determine the quality of the manufacture of the composite. These tag end tests included density, fiber content, and thermal analysis. After completing the tag end testing, the remaining sheet material was cut to the appropriate sample sizes and prepared for testing according to the following "initial preparation procedure".

Initial Composite Preparation Procedure

Testing specimens were dried in a vacuum oven at 63°C (145°F) over a weekend. The specimens were cooled to room temperature in a desiccator and weighed on an analytical balance to determine the "dry" weight. The specimens were then soaked in deicing fluid for four hours and air dried at room temperature for twenty hours. This process was repeated for four consecutive days, totaling twenty hours of immersion time per specimen. The specimens were then rinsed with DI water and allowed to remain at room temperature over the weekend. The exposed specimens were then reweighed to determine the total weight gain.

Mechanical testing (in-plane shear) of Specimen 1 was performed on Monday, Specimen 2 on Tuesday, and Specimen 3 on Wednesday.

Physical tests (Barcol indentation, glass transition temperature, sandwich corrosion, and thermal oxidative stability) were conducted as soon as possible after soaking.

4.2.2.1 Density/Specific Gravity

Test Description

Note: This was a tag end test procedure. Samples were not immersed prior to testing.

Four 1 x 1-inch specimens were cut from the PMC sheet. The temperature of the water in the immersion tank was measured and recorded. Each specimen was weighed in air to the nearest 0.1 mg, as well as the sinker. The specimen (and sinker) were placed in the sample holder and completely immersed in the tank, without contacting the sides of the tank. In addition, any bubbles attached to the sample holder, sample and sinker were removed. The weight was then recorded. Next, the weight of the sample holder and sinker, immersed in water at the same depth as the sample, was recorded. The density of the sample was then determined using the calculation listed in ASTM D 792. The density of the PMC sheet was the average of the density values for the four specimens per sheet.

Test Methodology

Parameters	1 x 1-inch sample measured on a balance equipped with a suspended sample holder and immersion tank	
Type/Number of Specimens	4 samples per PMC sheet	
Experimental Control Specimens	None	
Acceptance Criteria	Repeat density readings within $\pm 1\%$.	
Reference Document	ASTM D 792	
Test Equipment	Analytical Balance with sample suspension	

Test Results

The average density for the PMC material was determined to be 1.479 g/cm³ with a standard deviation of 0.007. For comparison, density of previously tested epoxy and bismaleimide (BMI) composites was 1.52 g/cm³ and 1.54 g/cm³, respectively.

4.2.2.2 Fiber Content

Test Description

Note: This was a tag end test procedure. Samples were not immersed prior to testing.

This method was used to determine the fiber content as percent by weight. The PMC specimens used for density analysis were each weighed to the nearest 0.1 mg and placed in separate beakers containing at least 30 millimeters of 70% nitric acid. Constant heat was applied with a hot plate up to 40° C (104° F). Once no trace of reinforcement/laminate combination remained, the contents of the beaker were vacuum filtered into a pre-weighed sintered glass filter or filter-lined crucible. The filter was washed with distilled water, then acetone, and placed into a pre-heated oven at 100° C (212° F) for approximately one hour. The filter was then cooled in a desiccator and weighed to the nearest 0.1 mg. The fiber content, in weight percent, was calculated by dividing the final mass of the specimen by the initial mass and multiplying by 100.

Test Methodology

Parameters	1 x 1-inch samples digested in nitric acid to	
	remove the laminate material	
Type/Number of Specimens	Samples (4) from density measurement (Section	
	4.2.2.1)	
Experimental Control Specimens	None	
Acceptance Criteria	NA	
Reference Document	ASTM D 3171	
Test Equipment	Vacuum pump	
	Analytical balance	

Test Results

The average fiber content for the PMC material was determined to be 76.86 % with a standard deviation of 4.22. Average fiber content for previously tested epoxy and BMI composite materials was 70.1% and 65.0%, respectively.

4.2.2.3 In-plane Shear

Test Description

This test was performed in triplicate. The tension test equipment was set up according to ASTM D 3039/D 3039M, where the testing was conducted with normal strain instrumentation in the longitudinal direction with continuous or nearly continuous load-normal strain data recording. Practice runs were performed, as necessary, to determine transducer placement, calibration needs, and optimum strain rate. The area of the specimen was measured at three places in the gauge section and reported as the average area for these determinations. The specimens were placed in the grips of the testing machine, taking care to align the long axis of the gripped specimen with the test direction. The grips were tightened and the pressures used on the grips recorded. Next, the transducers and strain-recording instrumentation were attached. The analyst recorded the load versus strain (or transducer displacement). The shear stress was calculated according to the formula provided in ASTM D 3518/D 3518M.

Test Methodology

Parameters	1 x 10-inch tensile test specimens of the laminate	
	sheets for each PMC which are tested with	
	extensometer to 5% shear strain.	
Type/Number of specimens	3 specimens	
Experimental Control Specimens	3 unexposed specimens	
Acceptance Criteria	Compare to unexposed sample results	
Reference Document	ASTM D 3518/D 3518M, ASTM D 3039/D 3039M	
Test Equipment	Tensile Test Machine	
	Extensometer – 1 inch	
	Extensometer Calibrator	

Test Results

The average shear stress and statistical analysis for the polymer matrix composite specimens for each material are listed in Table 9. Note that samples were also analyzed in Octaflo Type I PG deicer as a point of comparison, since this PMC materials is not the same as previous testing programs.

Table 9. Polymer Matrix Composite Shear Stress Test Results

Deicer	Sample	Shear Stress, ksi	Average Shear Stress
I In average d	1	91.2	90.8
Unexposed	2	90.4	90.8
Ostofia	1	88.1	91.2
Octaflo	2	94.2	91.2
EcoFlo	1	88.6	90.0
	2	89.4	89.0

All results for shear stress are within the range of error of each other, when comparing unexposed to each deicer. Therefore, there is no effect to shear stress from deicer exposure.

4.2.2.4 Glass Transition Temperature

Test Description

Note: This testing procedure was utilized for both tag end testing and physical testing after exposure.

This test method was used for initial tag end testing to determine composite quality, as well as physical testing after exposure to the deicing solutions. In both cases, a 1/8 x 1/8-inch specimen was cut from the sheet and dried in an oven at 93°C (200°F) until the sample weight loss was less than 0.01%. The specimen was then placed in a differential scanning calorimeter (DSC) at an appropriate temperature program and ramp rate to determine the onset temperature (glass transition temperature), or the point at which the sample begins to drastically change shape due to thermal expansion. This temperature point was recorded as the glass transition temperature and compared to standard values for tag end testing and unexposed specimen values from physical testing.

Test Methodology

Parameters	1/8 x 1/8-inch specimens, before (tag end) and after	
1 at afficters	_ · · · · · · · · · · · · · · · · · · ·	
	deicing solution exposure (physical test), were placed in	
	the DSC to determine the transition temperature	
Type/Number of Specimens	1 specimen (tag end); 2 specimens per deicing fluid	
	(physical tests)	
Experimental Control Specimens	2 unexposed specimens	
Acceptance Criteria	Compare to standard values (tag end testing) Compare to	
	unexposed sample results (physical testing)	
Reference Document	ASTM E 794	
Test Equipment	DSC	
	Analytical Balance	

Test Results

The glass transition results for the PMC specimens are listed in Table 10.

Table 10. Polymer Matrix Composite Glass Transition Temperature Results

Sample Description	Avg Glass Transition Temp (°C)
PMC DI Control - sample 1	Not detected
PMC Tag End - sample 1	Not detected
PMC unexposed Control - 1	Not detected
PMC unexposed Control - 2	53.1
PMC w/ Ecoflo – sample 1	Not detected
PMC w/ Ecoflo – sample 2	Not detected
PMC w/ Octaflo – sample 1	Not detected
PMC w/ Octaflo – sample 2	93.19

As seen in Table 10, the glass transition temperature could not be detected for most samples. The melting point was the only peak detected in these instances. Therefore, not knowing the composition of this composite material, the results of this test are inconclusive.

4.2.2.5 Barcol Hardness

Test Description

This test was used to determine the hardness of the PMC material before and after exposure to the deicing solutions. To conduct the test, the impressor and test specimen must be placed on a solid surface. The point sleeve was then placed on the surface to be tested and the legs of the impressor were placed on the same surface or on a solid material of the same thickness so that the impresser is perpendicular to the surface being tested. The instrument was grasped firmly and quick, uniformly increasing force applied on the case until the dial indication reached a maximum. The Barcol hardness number was then recorded from the dial. The test was repeated five times on the same specimen and the results averaged.

Test Methodology

Parameters	1 x 2-inch specimen
Type/Number of Specimens	2 specimens/5 tests per specimen
Experimental Control Specimens	2 unexposed specimens
Acceptance Criteria	Compare to unexposed sample results
Reference Document	ASTM D 2583
Test Equipment	Barcol Impressor

Test Results

Table 11 lists the results of the Barcol indentation test for each of the PMC samples. Each value provided for the samples is the average of five readings across the surface of the sample.

Table 11. Polymer Matrix Composite Material Barcol Indention Results

Sample Name	Average Hardness - before exposure	Average Hardness – after exposure	Percent change in hardness
Unexposed – 1	75.2	76.4	1.6 %
Unexposed - 2	76.0	77.2	1.6 %
EcoFlo – 1	76.0	75.4	0.8 %
EcoFlo – 2	76.6	75.8	1.0 %
Octaflo – 1	76.0	77.2	1.6 %
Octaflo - 2	75.6	76.6	1.3 %

The test results show that the deicing solutions had no effect on the hardness of the PMC material, generally having a change in hardness less than the unexposed samples.

4.2.2.6 Sandwich Corrosion

Test Description

The Humidity Test Cabinet was prepared as specified by ASTM D1748. Next, each test apparatus was constructed as a sandwich of the following materials: 1) a 2 x 4-inch sample of 2024-T3 aluminum, 2) a 1 x 3-inch piece of filter paper saturated with deicing fluid as the middle layer, and 3) a 2 x 4-inch section of polymer matrix composite material. All aluminum specimens had to be thoroughly cleaned to remove any oils or surface contaminants. The sandwich was then secured together and placed in a 100°C (212°F) oven for eight hours. The specimens were then placed in the humidity chamber at 38°C (100°F) with 95-100% humidity for 16 hours. This cycle was repeated for a total of five days. At the end of the fifth day, the samples remained in the humidity chamber for an additional 48 hours. Once the specimens were removed from the chamber, the sandwiches were opened, the panels were cleaned, and the filter paper discarded. Both the aluminum and polymer matrix composite portions of the sandwich were examined with a stereomicroscope to determine the severity of corrosion or discoloration. The rating scale from the ASTM is:

- 0—No visible corrosion and no discoloration present
- 1—Very slight corrosion or discoloration, and/or up to 5% of area corroded
- 2—Discoloration and/or up to 10% of area corroded
- 3—Discoloration and/or up to 25% of area corroded
- 4—Discoloration and/or more than 25% of area corroded, and/or pitting present

Test Methodology

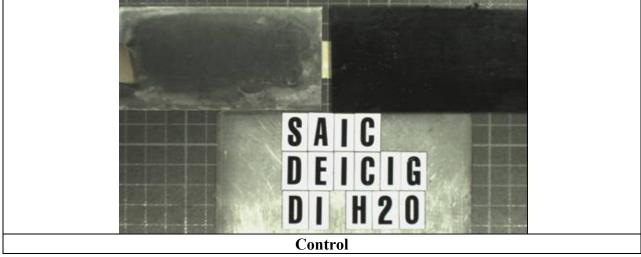
Parameters	Sandwich specimens consisting of 2024-T3 aluminum, filter paper saturated with deicing solution, polymer matrix composite specimens placed in 100°C (212°F) oven then humidity chamber set at 38°C (100°F)/95-100% humidity	
Type/Number of Specimens	Two specimens per PMC material	
Experimental Control Specimens	Two specimens exposed to DI water	
Acceptance Criteria	Compare to control specimens exposed to DI water	
Reference Document	ASTM F1110-90, ASTM D1748	
Test Equipment	Humidity/Corrosion Chamber	
	Drying Oven	
	Stereomicroscope	

Test Results

Table 12 contains the test results for the sandwich corrosion test, providing a rating and visual observations. Figure 3 shows a representative photo of the samples, with the aluminum panel on the left and the PMC portion of the sandwich on the right.

Table 12. Sandwich Corrosion Test Results

Sample	PMC Material		2024-T3 Aluminum		
Name	Rating	Comment	Rating	Comment	
Control 1	0	No corrosion	1	Slight edge corrosion	
Control 2	0	No corrosion	1	Slight edge corrosion	
EcoFlo – 1	0	Slight delamination, small blisters	1	Very slight edge corrosion	
EcoFlo – 2	0	Slight delamination, small blisters	1	Very slight edge corrosion	



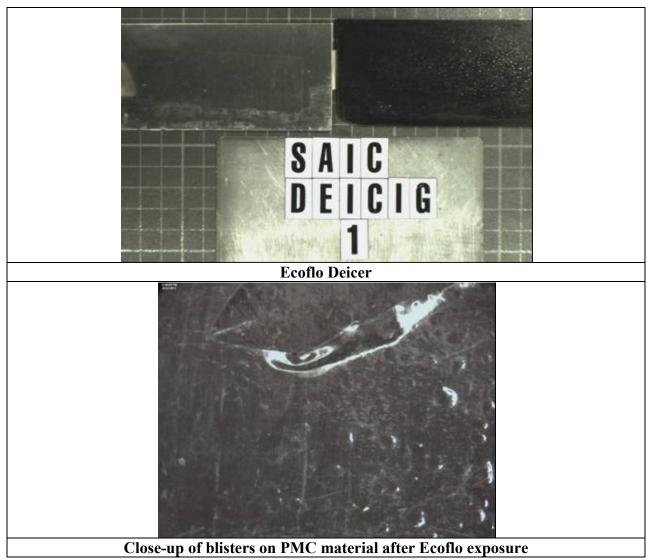


Figure 3. Photos of Sandwich Corrosion Samples

Test results for sandwich corrosion showed no discoloration or corrosion of the PMC material after exposure to deicer, indicating a passing rating. However, the PMC material did exhibit slight blistering, as seen in the close-up in Figure 3. The Type I deicer, Octaflo, was not evaluated with this PMC material, due to lack of material remaining.

4.2.2.7 Thermal Oxidative Stability

Test Description

Initial Preparation/Exposure:

Test specimens were dried in a vacuum oven at 145°F (62.8°C) over a weekend. The specimens were then cooled to room temperature in a desiccator and weighed on an analytical balance to determine the "dry" weights. The test specimens were then immersed in deicing fluid for four hours, then air dried at room temperature for twenty hours. This exposure procedure was

repeated daily for an additional four days, totaling twenty hours of immersion time per specimen. The test specimens were then rinsed with de-ionized water and allowed to dry at room temperature over the weekend. The exposed specimens were then weighed to determine the total weight gain, with additional testing being initiated as quickly as possible after this final weighing

Testing:

Thermal oxidative stability testing was performed on ten 1 x 1-inch samples of PMC material. The specimens were cleaned with Scotchbrite® (or equivalent) and soapy water and then submerged in an ultrasonic bath (water). The surface area of both sides was measured and the specimens were weighed to the nearest 0.1 mg. The specimens were then placed in a vacuum oven at 93°C (200°F) for at least 24 hours, and a small sample set was weighed. These samples were then placed back in the oven until the next day and reweighed. This process was continued until less than 0.01% weight loss is achieved. Once achieved, a final weight was recorded for each specimen. These weights were recorded as the "dry weights". Next, the specimens were placed on an oven rack in a convection oven that has been covered with a fiberglass breather cloth. The specimens were lightly covered with another layer of fiberglass breather cloth. The damper was configured to mostly closed (not air tight) and the oven heated to 204°C (400°F) at a rate of 4.7°C (10°F) per minute. The specimens were held at the maximum temperature for 100 hours. After 100 hours, the oven temperature was lowered to cool the specimens. The specimens were then placed in a desiccator to achieve room temperature. The specimens were again weighed with this value recorded as the final weight. The final weight was subtracted from the "dry weight" to determine the weight loss due to thermal exposure. Also, weight loss per surface area (milligrams per square centimeter [mg/cm²]) was calculated by dividing the weight loss by the specimen surface area.

Test Methodology

Parameters	1 x 1-inch PMC specimens exposed to deicer fluids, then heated in a convection oven at a ramp of 4.7°C (10°F) per minute to the maximum temperature for the material
Type/Number of Specimens	10 specimens per PMC material per deicing fluid
Experimental Control Specimens	10 specimens exposed to DI water
Acceptance Criteria	Compare to control specimens exposed to DI water
Reference Document	Draft MTMS
Test Equipment	Ultrasonic Bath
	Analytical Balance
	Drying Oven

Test Results

Table 13 lists the results of the Thermal Oxidative Stability test. Note that all data points are an average of the readings from ten samples. The standard deviation is also listed to show the level of repeatability of the measurements.

Table 13. Thermal Oxidative Stability Results

Sample Name	Total Weight Loss per		Average Weight	Standard
	Weight	Surface Area	Loss per	Deviation
	Loss (g)	(mg/cm^2)	Surface Area	
			(mg/cm ²)	
DI Control 1	0.0089	0.6622		
DI Control 2	0.0098	0.7051		
DI Control 3	0.0088	0.6259		
DI Control 4	I Control 4 0.0087 0.6203			
DI Control 5	0.0083	0.5970	0.59	0.07
DI Control 6	0.0068	0.4992	0.39	0.07
DI Control 7	0.0070	0.5115		
DI Control 8	0.0091	0.6577		
DI Control 9	0.0071	0.5073		
DI Control 10	0.0074	0.5449		
EcoFlo 1	0.0090	0.6393		
EcoFlo 2	0.0101	0.7249		
EcoFlo 3	0.0091	0.6514		
EcoFlo 4	Flo 4 0.0070 0.4998			
EcoFlo 5	0.0073	0.5177	0.60	0.07
EcoFlo 6	0.0085 0.6072		0.00	0.07
EcoFlo 7				
EcoFlo 8	0.0077	0.5775		
EcoFlo 9	0.0091	0.6509		
EcoFlo 10	0.0077	0.5650		
Octaflo 1	0.0116	0.8227		
Octaflo 2	0.0069	0.5251		
Octaflo 3	0.0086	0.6103		
Octaflo 4	0.0067	0.4851		
Octaflo 5	Octaflo 5 0.0071 0.5097		0.53	0.12
Octaflo 6	raflo 6 0.0054 0.3933		0.55	0.12
Octaflo 7	0.0069 0.4964			
Octaflo 8	0.0051	0.3748		
Octaflo 9	0.0072	0.5109		
Octaflo 10	0.0075	0.5331		

The average weight loss per surface area results for the PMC material exposed to DI water and each deicer were all within the standard deviation of each other, indicating no significant change in the PMC material from exposure to the deicing fluids.

4.2.2.8 Percent Weight Gain After Soak

Test Description

After the initial preparation procedure outlined in Section 4.2.2, each specimen that was used for testing was weighed to determine the final weight after soaking in the deicing fluid (or DI water). The "dry" weight (measured before the soak) was subtracted from the final weight and then multiplied by 100 to determine the percent weight gain.

Test Methodology

Parameters	All samples required for PMC tests soaked in the various	
	deicing fluids or DI water control	
Type/Number of Specimens	Test specimens for each PMC test method that are	
	exposed to deicer fluid according to the Initial Composite	
	Preparation Procedure	
Experimental Control Specimens	Test specimens exposed to DI water	
Acceptance Criteria	Compare to control specimens exposed to DI water	
Reference Document	JTP	
Test Equipment	Analytical Balance	

Test Results

Table 14 lists the averaged percent weight gain results for samples exposed to deicing fluid prior to additional testing. Note that percent weight gain was not calculated for the in-plane shear samples.

Table 14. Average Percent Weight Gain After Soak Results

Test Method	Fluid/Exposure	Average Percent Weight Gain (%)
	DI Water	0
Glass Transition	EcoFlo	0
	Octaflo	0
	Unexposed	0.12
Barcol Indentation	EcoFlo	0.11
	Octaflo	0.11
	DI Water	0.09
Thermal Oxidative Stability	EcoFlo	0.16
	Octaflo	0.08

The PMC samples showed similar weight gain from exposure to the test fluids compared with DI water or unexposed specimens, with the exception of the EcoFlo on the thermal oxidative stability test.

4.3 Elastomeric Materials

Elastomeric materials, which were comprised of sheet and bearing materials and sealants materials, have a wide variety of uses on aircraft and deicing trucks. These materials have been used to seal metal-to-metal contacts to discourage galvanic corrosion, act as a moisture or sound barrier, and provide heat or wear resistance, among other applications. The main effect that deicing material may have on elastomers is to cause brittleness from reducing the elastic properties of the material. The testing methods in this section were designed to assess and determine any changes in the elasticity of the materials.

4.3.1 Elastomeric Substrate Materials

The elastomeric materials identified included a total of two sheet materials and five sealants. These materials are listed in Table 15, along with the specification number and vendor where the material was purchased. The sheet materials were used as received for testing. The sealant materials were prepared in-house at the *CTC* test facilities, cured, and then cut into specimens. This procedure is further discussed in Section 4.3.2.

Substrate	Specification Number	Vendor
Nitrile Sheet	MIL-R-6855 Class I	Elastoseal, Inc.
Neoprene Sheet	MIL-R-6855 Class II	Elastoseal, Inc.
Polysulfide Sealant	MIL-S-8802 Type I	PRC DeSoto
High Temperature	AMS-3276C	PRC DeSoto
Polysulfide Sealant		
Corrosion-Inhibiting Sealant	MIL-PRF-81733D	PRC DeSoto
Polythioether Sealant	AMS-3277B	PRC DeSoto

Table 15. Elastomeric Materials

4.3.2 Elastomeric Materials Test Methods

Testing for Elastomeric materials included the following:

- 1. Shore A hardness ASTM D2240
- 2. Percent volume swell JTP
- 3. Peel strength and percent cohesive failure SAE AS 5127/1
- 4. Ultimate tensile strength and Percent Elongation SAE AS 5127/1
- 5. 100% and 300 % modulus SAE AS 5127/1

<u>Initial Specimen Preparation Procedures:</u>

Sealant Preparation

To prepare the sealant samples, the sealant kits were mixed according to manufacturer's recommendations. The sealant was then spread evenly onto a polyethylene sheet (or other suitable non-stick surface). A second polyethylene sheet containing spacers was then applied to

the sealant in order to sandwich the sealant to achieve the desired thickness of 0.25 inch. The sealant materials were then cured for the manufacturer's recommended cure time. At the end of the cure time, the polyethylene sheets were removed and the specimens were cut from the cured sealant.

Deicer Immersion

To prepare the specimens for testing (except percent volume swell testing), the nitrile and neoprene materials were immersed in the deicing solution for eight hours at room temperature then removed from the solution for sixteen hours at room temperature, with this cycle repeated four additional times. The sealant materials were immersed in the deicing solution for four hours at room temperature, then removed for twenty hours and this cycle was also repeated four additional times. All tests were performed on an unexposed set as well as the exposed material.

1.1.3.2.1 Shore A Hardness

<u>Test Description</u>

Shore A hardness measurements were conducted with a hand-held Shore Type A durometer. The durometer calibration was verified before beginning measurements by the use of a 60 duro test block. The durometer was placed on the specimen and the technician pressed down firmly on the durometer until the base rested on the sample. The hardness results were read from the gauge on the front of the durometer. The test was repeated three times and the average hardness result (HA) was reported for each specimen.

<u>Test Methodology</u>

Parameters	1 x 3-inch test specimens after deicer immersion; all	
	elastomeric materials	
Type/Number of Specimens	2 of each elastomeric material – 3 trials per specimen	
Experimental Control Specimens	2 unexposed samples of each elastomeric material	
Acceptance Criteria	Less than 10% difference in hardness value when	
	compared to unexposed sample.	
Reference Document	ASTM G-D2240	
Test Equipment	Type A Durometer	

Test Results

Table 16 lists the results of the Shore A Hardness testing for the Eco-Flo deicing solution. Pass or fail is listed in the far right hand column, based on the acceptance criteria that the hardness could not differ by more than 10% when compared to the unexposed hardness results. The standard deviation of the difference is also presented in the table, in order to determine the significance of the difference in the data. Table 17 contains the average change in hardness data to Type I PG Octaflo deicer from a previous test program, for comparison.

Table 16. Shore A Hardness Results

Deicer	Elastomer	Sample Number	% Change in Hardness (avg of 3 readings)	Avg. STD	Pass/Fail
	Nitrile	1	0.97	0.58	PASS
	Niune	2	1.46	0.58	PASS
	Neoprene	1	1.03	0.87	PASS
	Neopiene	2	0.51	0.58	PASS
	High Temp Polysulfide Sealant	1	2.86	1.16	PASS
EcoFlo		2	2.82	0.79	PASS
ECOFIO	Corrosion-	1	0.55	0.58	PASS
	Inhibiting Sealant	2	2.25	0.79	PASS
	Polysulfide	1	0.57	0.87	PASS
	Sealant	2	1.11	1.08	PASS
	Polythioether	1	1.60	1.05	PASS
	Sealant	2	3.21	0.87	PASS

Table 17. Average Hardness Results for Type I Octaflo Deicer (Previous Test Program)

Elastomer	Average Change in Hardness
Nitrile	3.11
Neoprene	3.29
High Temp Polysulfide Sealant	0.50
Corrosion-Inhibiting Sealant	3.03
Polysulfide Sealant	13.86
Polythioether Sealant	10.62

First, all elastomeric materials passed the test requirement of a less than 10% change in hardness due to exposure to the deicer. In addition, the EcoFlo deicer had lower changes in hardness than Type I Octaflo deicer for all elastomers, with the exception of the high temperature polysulfide sealant.

4.3.2.2 Percent Volume Swell

Test Description

Note: This test procedure was not associated with the initial sample preparation as mentioned above.

The samples for percent volume swell, 1 x 3-inch in size, were weighed on an analytical balance, while the dimensions were measured with calipers. The samples were placed in the deicing

solution for 72 hours at room temperature. Once the samples were removed and air dried, the samples were reweighed and the dimensions remeasured to determine if any swelling or shrinkage of the sample had occurred as a result of exposure to the deicing fluid. Results were reported as a positive or negative percent numerical change from the original sample weight or dimensions.

Test Methodology

Parameters	1 x 3-inch test samples after 72 hours of		
	immersion; all elastomeric materials		
Type/Number of specimens	2 of each elastomeric material		
Experimental Control Specimens	N/A		
Acceptance Criteria	Minimal (<1%) swell or shrinkage due to		
	anti-icing solution exposure		
Reference Document	Draft MTMS		
Test Equipment	Analytical Balance		
	Digital Micrometer		

Test Results

Table 18 lists the results of the percent weight gain and percent volume swell measurements. The pass/fail rating is listed to the right of the volume swell test results. As noted in the test methodology section, < 1% change in volume is considered a passing rating. There are no acceptance criteria listed for weight change.

Table 18. Percent Volume Swell and Percent Weight Change Results for EcoFlo Deicer

Deicer	Elastomer	Sample Number	% Change in Volume	Volume Avg. STD	Pass/Fail (Volume)	% Change in Weight
	Nitrile	1	2.36	0.012	FAIL	0.483
	Nitifie	2	1.49	0.005	FAIL	0.538
	Naopropo	1	1.31	0.006	FAIL	0.039
	Neoprene	2	5.34	0.006	FAIL	0.034
	High Temp	1	0.10	0.023	PASS	0.068
EcoFlo	Polysulfide Sealant	2	1.82	0.023	FAIL	0.104
ECOLIO	Corrosion-	1	4.76	0.062	FAIL	0.074
	Inhibiting Sealant	2	6.17	0.036	FAIL	0.105
	Polysulfide	1	10.37	0.012	FAIL	0.166
	Sealant	2	14.68	0.025	FAIL	0.088
	Polythioether	1	1.56	0.009	FAIL	0.190
	Sealant	2	0.74	0.006	PASS	0.235

The results in Table 18 show that the EcoFlo deicer caused volume changes of greater than one percent for at least one sample for each elastomeric type. The largest changes in volume occurred in corrosion-inhibiting sealant and polysulfide sealant, indicating that these materials were potentially absorbing the deicer. The changes in weight were less than one percent for all elastomeric materials. Table 19, below, lists the results for the Type I Octaflo deicer from a previous test program.

Table 19. Percent Volume Swell and Percent Weight Change for Octaflo Deicer

Elastomer	% Change in Volume	% Change in Weight
Nitrile	0.63	0.05
Neoprene	2.07	0.26
High Temp Polysulfide Sealant	0.41	0.06
Corrosion-Inhibiting Sealant	0.52	0.02
Polysulfide Sealant	2.01	0.13
Polythioether Sealant	8.06	0.17

The Octaflo deicer also exhibited failures in volume swell, with greater than 1% changes for neoprene, polysulfide, and polythioether.

4.3.2.3 Peel Strength and Percent Cohesive Failure

Test Description

This method applies to the sealant materials only; nitrile and neoprene were not tested. The sealants were prepared according to the manufacturer's instructions, sandwiching the material between a 2.75 x 5-inch piece of aluminum sheet (4045 aluminum alloy, sulfuric acid anodized, primed with MIL-PRF-23377 and coated with MIL-C-85285 topcoat) and wire mesh. After curing the specimens, one of two identical samples were immersed in deicing fluid according to the initial preparation procedure listed above. The other sample served as the baseline. After the exposure time specified in the initial preparation, the panels were allowed to remain in the respective deicing fluid for an additional day at standard conditions. The peel strength test was conducted within five minutes after removing panel from the deicing fluid.

To prepare the test specimens for the peel strength test, two, one-inch wide strips were cut through the wire mesh and sealing compound to the metal surface of the test panel and extended the full length of the wire mesh, creating two test sites per panel. The test specimen was then installed in a tensile test machine. The upper jaw was clamped to the test panel and the lower held the wire mesh. The wire mesh was stripped back at an angle of 180° to the metal panel at a jaw separation rate of two inches per minute. During the peel strength testing, three cuts were made through the sealant to the panel in an attempt to promote adhesive failure. The cuts were approximately one-inch intervals.

The results were reported as the numerical average of the peak loads during cohesive failure. Failure of the sealant compound to the wire mesh was included in the peel strength values.

Percent cohesive failure was simply a visual inspection of the peel strength panels after testing. A percentage rating was applied to the degree of cohesive failure between the sealant and the paint system, as well as between the paint system and the aluminum panel.

Test Methodology

Parameters	Sealant materials sandwiched between a coated aluminum panel and wire mesh/the panel and mesh are clamped into a tensile test machine and the wire mesh is pulled away at 180° angle		
Type/Number of Specimens	2 specimens per sealant		
Experimental Control Specimens	2 unexposed specimens per sealant		
Acceptance Criteria	Compare to unexposed specimen results		
Reference Document	Peel strength: SAE AS 5127/1		
	Percent cohesive failure: JTP		
Test Equipment	Tensile Test Machine		

Deviations from, or Interpretation of Test Method

Due to known adhesion issues with the preparation of samples in the past, two unexposed control panels were fabricated, along with two exposed panels, giving a total of four results for each condition. Also, 1826 Polythioether sealant material was not tested at the time of this report, due to long lead times on receiving the material. This information will be added at a later date.

Test Results

The results of the peel strength and percent cohesive failure are listed in Table 20 below. There are no previous results for the Type I Octaflo deicer for comparison, due to failures of the sample builds.

Table 20. Peel Strength and % Cohesive Failure

Sealant Material	Sample	Peak Load (Newtons)	Average Peak Load	% Cohesive Failure
1440, Polysulfide	Unexposed 1 A	158	170	32
	Unexposed 1 B	141		2
	Unexposed 2 A	160		22
	Unexposed 2 B	222		0
	EcoFlo 1 A	164	145	54
	EcoFlo 1 B	179		52
	EcoFlo 2 A	136		38
	EcoFlo 2 B	101		72
1750, High Temp Polysulfide	Unexposed 1 A	88		83
	Unexposed 1 B	133	128	32
	Unexposed 2 A	134		33
	Unexposed 2 B	157		0

	EcoFlo 1 A	111		60
	EcoFlo 1 B	112	128	62
	EcoFlo 2 A	139	120	33
	EcoFlo 2 B	151		20
870, Corrosion Inhibiting	Unexposed 1 A	126	134	17
	Unexposed 1 B	142		7
	Unexposed 2 A	146		4
	Unexposed 2 B	122		9
	EcoFlo 1 A	140		14
	EcoFlo 1 B	119	134	0
	EcoFlo 2 A	133	134	0
	EcoFlo 2 B	143		0

Results of the average peel strength show no effect from exposure to EcoFlo for high temperature polysulfide and corrosion-inhibiting sealants. The polysulfide had a drop in peel strength of about 15%.

Cohesive failure was a visual examination and determination of the percentage of failure that occurred within the sealant/wire system (cohesive), as opposed to adhesive failure of the sealant to the coating system. Cohesive failure is the preferred mode of failure in order to determine effect of deicer on the sealant material. From the results in Table 20, the polysulfide and high temperature polysulfide sealant both exhibited better cohesive failure percentages for the EcoFlo-exposed samples than the unexposed samples. The amount of cohesive failure was very low for both the unexposed and exposed samples for corrosion-inhibiting sealant.

4.3.2.4 Ultimate Tensile Strength and Percent Elongation

Test Description

These tests were utilized to determine the tensile strength of the specimen, in mega-pascals (MPa), and the percent elongation, which is the percent difference of the distance between the bench marks placed on the specimen before and after the sample was stretched in the tensile test machine. First, the specimens were cut from all of the elastomeric materials according to ASTM D 412, using Die C from Figure 2 as the example. The cross-sectional area of the specimen was determined in units of square inches. Bench marks were then secured to the specimen for percent elongation determination. The specimen was placed in the grips of the testing machine, using care to adjust the specimen symmetrically to distribute tension uniformly over the cross section. The rate of the grip separation was set to 20 ± 2 inches per minute. The force at the time of rupture was recorded and the extensometer was used to make the elongation measurement, measuring and recording the elongation to the nearest 10%. Tensile strength was calculated by using the force magnitude at rupture (mega-newtons [MN]) and the cross-sectional area of the unstrained specimen (square meter [m²]). The percent elongation was calculated by subtracting the original distance between the bench marks from the final distance of the extended specimen, dividing by the original distance, and then multiplying by 100.

Test Methodology

Parameters	All elastomeric materials prepared according to	
	ASTM D 412 then tested in a tensile machine at a	
	grip rate of 20 inches per minute	
Type/Number of Specimens	3 specimens of each elastomeric material	
Experimental Control Specimens 3 unexposed specimens of each elastomeric ma		
Acceptance Criteria	Compared with the sealing compound	
	specification/unexposed sample results	
Reference Document	SAE AS5127/1, ASTM D 412	
Test Equipment	Tensile Test Machine	

Test Results

Table 21 lists the results for the unexposed and EcoFlo deicers for UTS and percent elongation for each of the elastomer materials. Octaflo test results from a previous test program are also included for comparison.

Table 21. Ultimate Tensile Strength and Percent Elongation

Elastomeric	Sample	Tensile Strength	Percent
Material	Description	(MPa)	Elongation (%)
	Unexposed 1	13.9	450
	Unexposed 2	14.0	480
	Unexposed 3	13.6	460
Nitrile	EcoFlo 1	14.1	430
Nitifie	Ecoflo 2	14.3	450
	Ecoflo 3	14.2	450
	Octaflo (avg)	15.2	
		(16.0 – unexposed)	
	Unexposed 1	11.0	450
	Unexposed 2	11.6	480
	Unexposed 3	10.0	450
Naopropo	EcoFlo 1	10.9	440
Neoprene	EcoFlo 2	9.9	450
	EcoFlo 3	10.1	460
	Octaflo (avg)	12.5	
		(12.5 - unexposed)	
	Unexposed 1	3.4	340
	Unexposed 2	2.9	250
	Unexposed 3	3.0	270
Polysulfide	EcoFlo 1	3.1	290
	EcoFlo 2	3.1	280
	EcoFlo 3	3.1	280
	Octaflo (avg)	2.6	
		(2.8 – unexposed)	

	Unexposed 1	2.9	210
	Unexposed 2	3.3	250
		3.1	250
High-	Unexposed 3		
temperature	EcoFlo 1	3.0	230
Polysulfide	EcoFlo 2	3.0	230
Torysumae	EcoFlo 3	3.2	270
	Octaflo (avg)	3.0	
		(2.6 – unexposed)	
	Unexposed 1	3.8	230
	Unexposed 2	3.8	240
	Unexposed 3	3.6	190
Corrosion	EcoFlo 1	3.8	230
Inhibiting	EcoFlo 2	3.9	240
	EcoFlo 3	2.8	130
	Octaflo (avg)	2.1	
		(2.5 - unexposed)	
	Unexposed 1	3.3	120
	Unexposed 2	4.0	180
Polythioether	Unexposed 3	3.9	160
	EcoFlo 1	3.8	180
	EcoFlo 2	3.4	170
	EcoFlo 3	3.2	280
	Octaflo (avg)	2.6	
		(3.8- unexposed)	

The results for UTS indicate that the EcoFlo deicer had very little effect on the tensile strength of the elastomeric materials. In addition, the small changes that are noted are comparable to the previous results for the Type I Octaflo deicer. Also, the percent elongation results are within the standard deviation of the unexposed samples, indicating no effect from exposure to the EcoFlo deicer.

4.3.2.5 Modulus

Test Description

Modulus values were determined for all elastomeric materials utilizing the same Die C-shaped specimen as the UTS and percent elongation measurements. The specimens were placed in the tensile test machine with the grip rate set at 20 ± 2 inches per minute. The machine recorded the force required to stretch the specimen in graphical form, with 100% and, if possible, 300% modulus determined from the load versus extension curves.

Test Methodology

Parameters	All elastomeric materials stretched in the same tensile

	set up as the UTS to 100% and possibly 300% extension
Type/Number of Specimens	3 specimens of each elastomeric material per deicing
	fluid
Experimental Control	3 unexposed specimens of each elastomeric material
Specimens	and 3 specimens exposed to DI water
Acceptance Criteria	Compared with DI water-exposed control
Reference Document	SAE AS5127/1
Test Equipment	Tensile Test Machine

Test Results

Table 22 lists the results for the 100% and 300% modulus testing. Results are reported as the UTS at 100% elongation and 300% elongation. In some instances, the samples broke before attaining 300% elongation, and are listed in the table as <300%.

Table 22. 100% and 300% Modulus Results

Elastomeric	Sample	UTS at 100%	UTS at 300%
Material	Description	(MPa)	(MPa)
	Unexposed 1	14.5	13.4
	Unexposed 2	14.3	13.5
	Unexposed 3	13.9	14.2
	DI Control 1	13.3	13.1
Nitrile	DI Control 2	13.5	13.9
	DI Control 3	13.6	13.8
	EcoFlo 1	14.3	14.4
	EcoFlo 2	12.4	14.3
	EcoFlo 3	14.3	14.4
	Unexposed 1	10.5	10.8
	Unexposed 2	9.9	10.7
	Unexposed 3	9.0	9.6
	DI Control 1	9.6	9.9
Neoprene	DI Control 2	10.2	10.4
	DI Control 3	8.9	10.0
	EcoFlo 1	10.7	9.6
	EcoFlo 2	9.5	10.5
	EcoFlo 3	10.8	10.4
	Unexposed 1	3.1	3.0
	Unexposed 2	2.2	3.5
	Unexposed 3	3.0	< 300%
Polysulfide	DI Control 1	2.5	< 300%
	DI Control 2	3.0	2.8
	DI Control 3	2.8	< 300%
	Ecoflo 1	3.3	3.4

	Ecoflo 2	3.2	2.6
	Ecoflo 3	3.1	3.1
<u> </u>	Unexposed 1	1.9	Not tested
	Unexposed 2	3.4	Not tested
	Unexposed 3	3.1	Not tested
High-	DI Control 1	3.4	3.1
temperature	DI Control 2	3.5	3.2
Polysulfide	DI Control 3	2.4	3.4
	EcoFlo 1	3.4	3.0
	EcoFlo 2	3.5	3.4
	EcoFlo 3	3.8	3.4
	Unexposed 1	4.0	< 300%
	Unexposed 2	3.9	< 300%
	Unexposed 3	2.6	<300%
Corrosion	DI Control 1	3.3	4.2
	DI Control 2	3.1	3.5
Inhibiting	DI Control 3	3.4	4.1
	EcoFlo 1	3.6	<300%
	EcoFlo 2	3.8	3.9
	EcoFlo 3	3.8	3.7
	Unexposed 1	2.7	<300%
	Unexposed 2	3.1	<300%
Polythioether	Unexposed 3	2.8	3.3
	DI Control 1	4.5	<300%
	DI Control 2	4.5	4.2
	DI Control 3	4.4	3.2
	EcoFlo 1	3.8	3.8
	EcoFlo 2	3.7	4.1
	EcoFlo 3	3.9	3.9

The results of the modulus testing showed consistent values for UTS when compared to the UTS testing results from Section 4.3.2.4. The nitrile and neoprene sheets materials had the highest UTS values, and the sealants all had values in the 2-5 MPa range. Again, there appears to be little effect on the elastomeric materials from exposure to EcoFlo deicer. In some instances, the tensile strength increased from the unexposed values.

4.4 Aircraft Wire Insulation

The following section describes the materials and testing methods that were used to assess the effects of the EcoFlo deicer on aircraft wire insulation. Both physical integrity of the wire was tested as well as functionality after exposure.

4.4.1 Aircraft Wire Insulation Materials

Aircraft wire insulation materials were required to evaluate the effects of the deicing material on the wire insulation. Electrical defects due to insulation breakdown, causing shorts or arcs, also were determined. Four types of insulation materials were tested. Table 23 contains a list of the materials, the specification number, and the vendor of the product.

Table 23. Aircraft Wire Insulation Materials

Substrate	Specification Number	Supplier
Polyimide	MIL-W-81381/11-20	Whitmor/Wirenetics
Teflon	MIL-W-22759/11-20	Whitmor/Wirenetics
Hybrid Construction	MIL-W-22759/86-20	Whitmor/Wirenetics
Cable-insulated Twisted Pair	MIL-W-22759	Whitmor/Wirenetics

4.4.2 Aircraft Wire Insulation Test Methods

The aircraft wire insulation was tested by the following procedures:

- 1. Conductivity JTP
- 2. Immersion test SAE AS 4373, Test Method 601
- 3. Bend test for post immersion cracking sensitivity SAE AS 4373 Test Method 714
- 4. Voltage withstand test SAE AS 4373, Test Method 5.10, ASTM D3032

4.4.2.1 Conductivity

Test Description

Conductivity measurements were performed on the deicing fluid and a DI water blank. The meter was calibrated with the appropriate standard solution and the conductivity of each deicing fluid was measured three times and averaged. The conductivity cell was thoroughly cleaned between each sample reading by rinsing with hot water and then DI water until the DI water conductivity values are achieved.

Test Methodology

Parameters	Conductivity measurements of the deicing	
	solutions with digital conductivity meter	
Type/Number of Specimens	Three samples of deicing solution	
Experimental Control Specimen	DI water	
Acceptance Criteria	Relative standard deviation (RSD) of the	
	three results is less than 2%	
Reference Document	JTP	
Test Equipment	Conductivity meter	

Results and Discussion

Table 24 lists the results of the conductivity analysis for the anti-icing fluids.

Table 24. Deicing Fluid Conductivity Results

Test Fluid	Sample No.	Conductivity µS
	1	<2
DI Water	2	<2
	3	<2
Average		<2
	1	230
EcoFlo Deicer	2	233
	3	232
Average		232

The EcoFlo had significantly higher conductivity than the DI water standard.

4.4.2.2 Immersion Test

Test Description

Four 24-inch lengths of each wire type were used for testing: three for immersion, and one to serve as a control. The outside diameter of each wire was measured in three places with digital calipers. Next, the wires were submerged in deicing fluid to within six inches of each end with the radius of the bend in the wire being between 14 and 35 times the maximum diameter of the wire being tested. The wires remained submerged in the test solution for 140 hours at room temperature, and the deicing solution was stirred several times throughout the test period. After 140 hours, the wires were removed from the test solution, washed with DI water, and dried at room temperature. The diameters of the wires were re-measured and any physical changes to the insulation were noted. The wires were then placed back into the deicing solution for an additional 140 hours. Again, after the exposure time, the wires were removed, washed, and dried at room temperature. The diameters were measured once more and any additional effects from deicer exposure were noted.

Test Methodology

Parameters	24-inch length of each wire type submerged in the	
	deicing solution; measured diameter with digital calipers	
Type/Number of Specimens	3 specimens per wire type	
Experimental Control Specimens	1 unexposed specimen	
Acceptance Criteria	Compared to unexposed control specimen	
Reference Document	SAE AS 4373, Test Method 601	
Test Equipment	Digital Micrometer	
	Stereomicroscope	

Results and Discussion

Table 25 lists the average change in diameter for each wire type.

Table 25. Wire Immersion Results

Wire Type	Fluid	Average % Change in Diameter
Polyimide	EcoFlo	-2.99
Teflon	EcoFlo	-0.54
Hybrid Construction	EcoFlo	-0.31
Cable Insulated Twisted Pair	EcoFlo	-0.12

The wires did not exhibit significant change in diameter when exposed to the deicing fluid. All but one of the wire types exposed to the deicing fluid experienced diameter changes of less than 1%. The one type was the polyimide wire, which had an average change in diameter of about 3%.

4.4.2.3 Bend Test for Post Immersion Cracking Sensitivity

Test Description

Following the immersion test, each wire was subjected to the bend test. A one-pound weight was attached to the end of the 20-gauge wire sections and the other end of the wire was wound over a 1.27-inch diameter mandrel. For the cable containing two 22-gauge wires, a 1.60-inch diameter mandrel and a two-pound weight was used. The wire was wound and then rewound in the reverse direction. The winding sequence was repeated a second time, so that two bends were formed in each direction in the same section of wire. The wire was visually inspected for damage to the insulation (include digital photos or stereomicroscope photos where necessary).

Test Methodology

Parameters	24-inch length of each wire from the immersion test
Type/Number of Specimens	3 specimens per wire type
Experimental Control Specimens	N/A
Acceptance Criteria	No visual damage to the insulation
Reference Document	SAE AS 4373, Test Method 714
Test Equipment	Mandrel
	Stereomicroscope
	Weights

Test Results

All test specimens passed the acceptance criteria for this test with no signs of cracking or breaking of the insulation. The EcoFlo deicing fluid did not adversely affect the integrity of the

aircraft wire materials. The wire coatings remained intact and visual inspections were performed to note observations prior to voltage withstand testing.

4.4.2.4 Voltage Withstand Test

Test Description

This test was performed after the immersion and bend tests, with the same wire samples. The purpose was to determine the integrity of the insulation following immersion and bend testing. The insulation was stripped from the last inch of wire on both ends and the ends were twisted together. Next, the wire was soaked in a salt solution (5% sodium chloride and 0.1% Triton X-100 wetting agent) for a minimum of four hours. Using a high potential (HIPOT) tester, voltage was applied between the twisted ends of the conductor and the grounded solution bath. The voltage was increased from 0 to 2200 volts – AC current (VAC) at a rate of 500 volts per second (V/s) with the peak voltage applied for one minute.

Test Methodology

Parameters	Four 24-inch length of each wire from the immersion	
	and bend tests	
Type/Number of Specimens	3 specimens per wire type	
Experimental Control Specimen	1 unexposed specimen	
Acceptance Criteria	Performs as well as control specimen	
Reference Document	SAE AS 4373, Test Method 510	
Test Equipment	HIPOT tester	

Test Results

Table 26 lists the results of the voltage withstand testing for each wire type.

Table 26. Dielectric Voltage Withstand Results

Wire Material	Sample ID	Current Measurements (mA)	Results
	Unexposed	0.21	PASS
Dolvimido Wiro	EcoFlo 1	0.21	PASS
Polyimide Wire	EcoFlo 2	0.21	PASS
	EcoFlo 3	0.21	PASS
	Unexposed	0.15	PASS
Teflon Wire	EcoFlo 1	0.17	PASS
Terion wire	EcoFlo 2	0.16	PASS
	EcoFlo 3	0.16	PASS
Habrid Construction	Unexposed	0.19	PASS
Hybrid Construction Wire	EcoFlo 1	0.19	PASS
w ire	EcoFlo 2	0.20	PASS

	EcoFlo 3	0.20	PASS
	Unexposed	0.37	PASS
Cable-Insulated	EcoFlo 1	0.37	PASS
Twisted Pair	EcoFlo 2	0.39	PASS
	EcoFlo 3	0.37	PASS

The voltage withstand testing did not result in any failures or leakage due to insulation breakdown. All wire exceeded the rigorous testing conditions performed.

4.5 Carbon-carbon Brake Friction Materials

There has been a long-standing complaint that deicing fluids cause significant damage to aircraft braking materials. This section of testing assessed the effects of the test deicing solutions on "pucks" cut from these brake materials. The test method that was developed for this purpose attempted to simulate brake conditions on an aircraft, where the materials were exposed to deicing solution, then to an intense heating cycle.

4.5.1 Carbon-carbon Brake Friction Material Specimens

CTC procured this material from Honeywell, an OEM of stators and rotors. The vendor cut 2-inch diameter specimens from random normal-production parts and coated half of the lot with their typical production anti-oxidant coating. Due to lack of material, *CTC*, with approval from SAIC, then cut these specimens in half. The specific Honeywell carbon/carbon materials are:

- CARBENIX 1000
- CARBENIX 2000
- CARBENIX 2330
- CARBENIX 4000

4.5.2 Carbon-carbon Brake Friction Material Test Method - Oxidation Resistance (Cyclic Heating and Shore D Hardness)

Test Description

This procedure was used to verify the effects of the deicing fluid on carbon-carbon composite aircraft brake friction materials, specifically determining the rate and extent of oxidation. Test samples were prepared by cutting 2-inch diameter specimens from rotors or stators using a carbide tipped saw. Ten specimens were cut for each type of carbon-carbon material, with five of the specimens brushed with anti-oxidant coating and cured. Upon receipt, *CTC* then cut these specimens in half (with SAIC approval) in order to accommodate performing control testing. The initial sample weight and hardness was measured for each specimen, then each set was immersed in deicing fluid or DI water (controls) for twenty minutes. The specimens were then removed from solution and dried at 43°C (110°F) for thirty minutes. The contaminated weight and hardness were measured after cooling. Next, the first heating cycle was completed by placing the sample sets in a preheated oven at 704°C (1300°F) for four hours. The sets were immediately removed from the oven and cooled in laboratory ambient air- the specimens are not actively cooled. The first heat cycle weight and hardness were then measured. The specimen

sets were then placed back into the oven at 704°C (1300°F) for an additional four hours. The final heat cycle weight and hardness were measured after ambient air-cooling.

Test Methodology

Parameters	2-inch diameter samples, cut in half, of carbon-carbon brake friction materials were immersed in deicing solution then heated in cycles in a 704°C (1300°F) oven
Type/Number of Specimens	10 specimens of each material (5 with and 5 without anti-oxidant coating)
Experimental Control Specimens	10 specimens of each material in DI water (5 with and 5 without anti-oxidant coating)
Acceptance Criteria	Performs as well as control specimen
Reference Document	JTP
Test Equipment	Type D Durometer
	Analytical Balance
	Muffle Furnace

Deviations from Test Method

The vendor provided 2-inch diameter samples for testing; however, upon receipt there were not enough to complete all testing. Therefore, with SAIC approval, all 2-inch samples were cut in half through the diameter to produce 2 semi-circle samples. These semi-circles were utilized for testing per the Test Methodology noted above.

Test Results

Table 27 contains the average percent weight loss and average percent hardness loss for the carbon-carbon brake friction materials that do not contain an anti-oxidant coating.

Table 27. Carbon-Carbon Break Oxidative Resistance Test Results of Uncoated Samples

Carbon-Carbon Brake Friction Material / Fluid	Average Weight		ce from DI Control	Average Hardness	Difference Water C	
	Loss, %	Weight	STD	Loss, %	Hardness	STD
		Loss %			Loss %	
CARBENIX 1000						
DI Water	60.96	STD =	2.356511	71.38	STD =	3.973787
EcoFlo	49.84	-11.12	-4.718131	38.02	-33.36	-8.39586
CARBENIX 2000						
DI Water	16.36	STD =	1.828481	23.06	STD =	4.817627
EcoFlo	35.57	19.21	10.50576	14.96	-8.11	-1.6827
CARBENIX 2330		•				
DI Water	24.89	STD =	0.68402	31.06	STD =	8.771043

EcoFlo	47.25	22.36	32.68559	61.91	30.85	3.516769
CARBENIX 4000						
DI Water	8.90	STD =	2.582676	7.45	STD =	5.088996
EcoFlo	49.96	41.06	15.89775	55.08	47.62	9.357714

Table 28 contains the average percent weight loss and average percent hardness loss for the carbon-carbon brake friction materials with anti-oxidant coating.

Table 28. Carbon-Carbon Brake Oxidative Resistance Test Results of Coated Samples

Carbon-Carbon Brake Friction Material / Fluid	Average Weight			Average Hardness	Difference Water C	
	Loss, %	Weight	STD	Loss, %	Hardness	STD
		Loss %			Loss %	
CARBENIX 1000						
DI Water	11.57	STD =	2.8225	7.30	STD =	4.894834
EcoFlo	8.37	-3.20	-1.134234	5.60	-1.70	-0.34642
CARBENIX 2000						
DI Water	5.64	STD =	10.74422	9.56	STD =	8.836085
EcoFlo	5.88	0.24	0.022327	3.94	-5.62	-0.63556
CARBENIX 2330						
DI Water	2.27	STD =	0.436849	-0.30	STD =	6.74997
EcoFlo	1.48	-0.79	-1.815251	-5.33	-5.03	-0.74486
CARBENIX 4000						·
DI Water	2.45	STD =	20.62754	-6.66	STD =	26.82348
EcoFlo	1.56	-0.89	-0.043314	-8.55	-1.89	-0.07033

<u>Uncoated Brake Material:</u> The uncoated brake materials showed noticeable trends in oxidation. The DI water control samples, for the 2000, 2330, and 4000 material, showed a lower weight loss when compared to the deicer exposed samples of the respective material. However, 1000 material DI water control sample showed a greater weight loss when compared to the deicer exposed material. The DI water sample for 1000 material exhibited the greatest hardness loss for test samples. The deicer exposed samples exhibited a percent loss of hardness ranging from 14.96 – 61.91 %. These extreme losses in weight and hardness are typical for uncoated samples.

Coated Brake Material: The coated brake materials showed some trends in oxidation, as well. The coated 1000, 2330, and 4000 brake materials exposed to the deicer fluid exhibited lower weight loss then the DI water exposed control samples, while the 2000 material exhibited a greater weight loss of for the deicer-exposed sample when compared to its control sample. The deicer-exposed samples for the 1000 and 2000 material exhibited a lower loss of hardness while the deicer-exposed samples 2330 and 4000 materials exhibited a greater loss in hardness. Again, these ranges for weight and hardness loss are typical for the oxidative resistance of the coated samples.

4.6 Infrared Window Material

This section describes the materials and testing method that was used to assess the effects of EcoFlo deicer on IR window materials. The change in IR transmission through the material or reflectance was measured before and after exposure.

4.6.1 Infrared Window Material Specimens

Infrared window material was procured from reputable vendors. The test specimens were one-inch diameter lenses with a standard commercial polish and thickness. The specific infrared windows and vendors are listed in Table 29.

Table 29. Infrared Window Materials

Substrate	Vendor
ALON	Surmet Corporation
Sapphire	Crystal Systems

4.6.2 Infrared Window Material Test Method - Fourier Transform Infrared (FTIR) Transmission

Test Description

This test was used to determine if the deicing fluid damaged IR window materials by measuring the transmission through the windows after exposure to the deicing fluid. Two 1-inch diameter disks were procured for the deicing fluid being tested. Using FTIR, pre test measurements were performed on sapphire consisting of IR transmission spectra in the range of 8-11.5 microns. For the ALON material, the transmission spectra were in the range of 3-5 microns. Pre-test visual exams of each material was performed and photographs were taken (digital camera or stereomicroscope) if necessary. Separate samples were immersed in deicing fluid for a cycle of 4 hours immersion and 20 hours drying, for a five-day period. After removal from the deicing fluid for the last time, each sample was washed with DI water and dried with nitrogen gas. Post-test IR transmission spectra measurements were made, reporting extra transmission peaks or loss of transmission. Any visual staining, discoloration, or clouding of the window materials was also reported and photos were taken, as necessary.

Test Methodology

Parameters	1-inch diameter disks, immersed in deicing solution and	
	measured before and after immersion for IR transmission	
Type/Number of Specimens	2 specimens of each window material	
Experimental Control	2 specimens of each window material immersed in DI water	
Specimen		
Acceptance Criteria	Staining or discoloration of material surfaces shall not	
	exceed that which can be cleaned with water, acetone	
	alcohol, or similar solvents and transmission loss due to	

	exposure shall not exceed 10%	
Reference Document	JTP	
Test Equipment	FTIR	
	Stereomicroscope	

Test Results

Change in infrared transmission and any visual changes to the windows due to deicer exposure are listed in Tables 30 and 31.

Table 30. Infrared Window Change in Transmission Results

Window Material	Deicer	Change in % Transmission (Thunderdome)	Change in % Transmission (PIKE Sample Clip)
	Control 1	+2.23	+2.34
ALON	Control 2	+3.83	+2.45
	Sample 1	-0.22	+2.42
	Sample 2	-3.82	+2.46
	Control 1	+4.69	0.00
Sapphire	Control 2	+0.59	0.00
	Sample 1	+2.61	0.00
	Sample 2	+1.60	+0.01

Table 31. Visual/Stereomicroscope Observations of Post-Immersion Windows

Window	Deicer	Visual Observations			
Material	Deicei	Staining	Discoloration	Clouding	Scratches
	Control 1	0	0	0	0
ALON	Control 2	0	0	0	0
	Sample 1	0	0	0	0
	Sample 2	0	0	0	0
	Control 1	0	0	0	0
Sapphire	Control 2	0	0	0	0
	Sample 1	0	0	0	0
	Sample 2	0	0	0	0

The results in Table 30 show that the deicer had minimal effect the transmission of the window materials, performing within the acceptance criteria limit of 10%. There was no discoloration, staining, clouding or scratching of the window materials, as noted in Table 31.

4.7 LO Coatings

LO coatings are more recent formulations of the aircraft paint systems that have been in use for decades. Exposure to deicing materials may cause loss of adhesion or softening of the coating.

This series of material compatibility testing has been designed to determine the effects of deicing materials on two paint systems applied to three substrate materials.

4.7.1 LO Coating Materials and Substrates

CTC procured the coatings from PRC DeSoto, Deft, and CAAP CO, with the coatings and vendors listed in Table 32.

Substrate

MS-133 Outer mold line primer

MS-424 Inner mold line primer

MS-484 Anti-static Rain Erosion
Urethane Coat

MS-485 Rain Erosion Urethane Coat

CAAP CO

CAAP CO

Table 32. LO Coatings

The coatings were applied to the following substrates, either as a primer-topcoat system or as a single coating, depending on the testing purpose:

- 1. 7075-T6 Aluminum alloy, AMS 4045H
- 2. 4140 steel, AMS 6395
- 3. AZ91E-T6 magnesium AMS 4446A

Paint adhesion testing was conducted in two parts. First, the primers only were applied to each substrate material to test the effects of the EcoFlo deicer on primer-substrate adhesion. Next, topcoats were applied over the primers on the 7075-T6 aluminum alloy only, to test deicer effect on intercoat adhesion. Hardness was also conducted on the primer-topcoat systems. Finally, liquid uptake testing was performed on each coating separately when applied to the 7075-T6 aluminum.

4.7.2 LO Coating Testing Methods

The following test methods of adhesion (2 types), hardness, and liquid uptake were performed to determine the integrity of the various coating-substrate combinations when exposed to the deicing materials. Section 4.7.2.1 describes the test panel preparation and coating application procedures to prepare the panels for testing.

4.7.2.1 Test Panel Preparation

Test Description

This test method describes the paint preparation and then immersion of the coatings in the deicing fluid, with DI water as the control, to prepare for testing. First, each 3 x 6 x 0.02- inch panel was wiped with acetone, dried and then lightly abraded with a fine aluminum oxide mat. The surface was then rinsed with distilled water and dried. The aluminum panels were

conversion coated according to MIL-C-81706, Class 1A. Following manufacturer's recommendations, each coating system was applied as appropriate to perform the testing procedures outlined in Sections 4.7.2.2 to 4.7.2.4. Each coating system was also dried according to the instructions provided by the manufacturer. Dry film thickness (ASTM D7091) and gloss (ASTM D523) measurements were recorded after drying.

4.7.2.2 Liquid Uptake

Test Description

This test method was used to determine the relative rate of absorption of deicing materials by the test coatings. Each coating was applied as a single coating to the conversion-coated aluminum substrates, and was immersed for 30 minutes or 2 hours in the deicing solution. To conduct the testing, a coated test panel that had been fully cured was measured to four decimal places on an analytical balance. The test specimen was then fully immersed in the deicing solution that was maintained at a temperature of $23 \pm 1^{\circ}\text{C}$ ($73 \pm 3^{\circ}\text{F}$). Half of the test panels were removed from the deicing solution after 30 minutes of exposure while the other half of the specimens remained exposed for 2 hours. After removal from the solution, the panels were rinsed with DI water, dried with a lint-free cloth (ensuring all surface water is removed), and reweighed. The percent change in weight was then calculated for each specimen. In addition, gloss measurements were taken and results of visual examination of the panels were recorded.

Test Methodology

Parameters	Weighed and completely immersed 3 x 6 x 0.02 inch
	cured test coatings in anti-icing solution for 30 minutes
	or two hours, panels were then removed, wiped dry, and
	reweighed. Measured gloss and performed visual exam.
Type/Number of Specimens	3 specimens of each coating type per deicing solution on
	7075-T6 Al substrate only
Experimental Control Specimen	3 specimens of each coating on 7075-T6 Al substrate
	only exposed to DI water
Acceptance Criteria	Percent change in weight equivalent to DI water
Reference Document	ASTM D 570, ASTM D7091
Test Equipment	Analytical Balance
	Drying Oven

Test Results

Table 33 lists the results of the liquid uptake test as average change in weight for the three panels tested, as well as average change in gloss.

Table 33. Liquid Uptake Results

			30 Minutes of Exposure			2 Hours of Exposure		
Coating	Fluid	Average Change in Weight	Average Change in Gloss at 60°	Average Change in Gloss at 85°	Average Change in Weight	Average Change in Gloss at 60°	Average Change in Gloss at 85°	
Outer	DI Water	0.02%	-0.59%	0.83%	-0.01%	-0.06%	0.25%	
Mold Line Primer	EcoFlo	0.02%	0.01%	0.83%	0.01%	-0.23%	-0.04%	
Inner	DI Water	0.05%	0.43%	0.88%	-0.03%	-0.25%	0.05%	
Mold Line Primer	EcoFlo	0.04%	0.37%	0.86%	0.06%	0.02%	0.25%	
Anti-	DI Water	0.07%	-0.01%	0.04%	-0.01%	0.54%	-0.08%	
Static Rain Erosion Topcoat	EcoFlo	0.05%	0.0%	0.09%	0.06%	-0.01%	-0.05%	
Rain	DI Water	0.05%	0.07%	0.32%	-0.01%	0.32%	0.01%	
Erosion Topcoat	EcoFlo	0.07%	0.21%	0.44%	0.10%	0.30%	0.28%	

The EcoFlo deicing fluid had no significant effect on the weight change of the four coatings when compared to DI water at 30 minutes exposure or the outer mold line primer at 2 hours exposure. Slightly higher weight change was observed compared with DI water at 2 hours exposure for the inner mold line primer, anti-static rain erosion, and rain erosion coatings, with the rain erosion topcoat showing the greatest change of 0.10%.

Overall, the change in gloss readings was also comparable to DI water-exposed samples at 30 minutes and 2 hours of exposure. And, to summarize, all changes in weight and gloss readings were less than 1.0%, with EcoFlo-exposed sample results equivalent to the DI water-exposed results.

4.7.2.3 Adhesion

<u>Test Description</u>

This test method covers procedures for deicing exposure and assessing the adhesion of the coating films to the metallic substrates by applying and removing pressure-sensitive tape over cuts made in the film. Two types of adhesion testing were performed: Method A (modified) and Method B. Both test methods were used to assess substrate adhesion and intercoat adhesion of

the coating with and without exposure to the deicing solution. Each test method is described below.

Immersion - After the paint drying cycle, each test panel was placed in a horizontal position in an oven maintained at $38 \pm 2^{\circ}$ C ($100 \pm 5^{\circ}$ F). The deicing solution was applied to approximately one half the area of each panel and allowed to remain on the panel for 30 minutes. Then, the panels were removed from the oven, rinsed with DI water, and allowed to air dry for 24 hours. Each panel was then visually examined for streaking, discoloration, or blistering of the finish prior to adhesion testing.

Test Method A – After immersion, an area on each half of the panel was selected for testing that was free of blemishes and minor surface imperfections. Two cuts were made in the film at each area, about 1.5 inches long, that intersect near their middle with a smaller angle between 30° and 44°. Then, two parallel lines were made through the "X", away from the center intersection. Two complete laps of the pressure-sensitive tape were removed from the roll and discarded. An additional length was then removed at a steady rate and a 3-inch piece was cut for use. The tape was affixed to the cuts in the coating by placing the center of the tape at the intersection of the cuts with the tape running in the same direction as the smaller angles. The tape was smoothed into place in the area of the incisions and then rubbed firmly with an eraser on the end of a pencil. Within 90 ± 30 seconds of application, the tape was removed by seizing the free end and pulling it off rapidly (not jerking) back upon itself at as close to an angle of 180° as possible. The incisions were inspected for removal of coating from substrate or previous coating. The extent of coating removal was then rated on the 0 to 5 scale outlined in the ASTM Standard.

Test Method B – After immersion, an area on each half of the panel was selected for testing that was free of blemishes and minor surface imperfections. For coatings having a dry film thickness up to and including 2.0 mils, cuts in a lattice design were spaced 1 mm apart, with 11 cuts total. For coatings having a dry film thickness of 2.0 mils to 5.0 mils, the cuts were spaced 2 mm apart, with six cuts to make up the lattice. All cuts were 1.4 inches long. The film was cut through to the substrate in one steady motion using just sufficient pressure on the cutting tool to have the cutting edge reach the substrate. After making the required cuts, the film was lightly brushed with a soft brush to remove any detached flakes or ribbons of coatings. The incisions were inspected for reflection of light from the substrate. The tape was then applied and removed in the same manner as described in Method "A". After removing the tape, the lattice was visually inspected for removal of the coating, with the analyst making note of any coating removal and rating the percentage of removal on the 0 to 5 scale.

Test Methodology

Parameters	Made appropriate cuts in the exposed and unexposed portions of the 3 x 6 inch coated panels, applied and removed tape, evaluated coating removal
Type/Number of Specimens	3 panels per coating system (primer only on 4 substrates – do not apply classic organic primers to composite substrate, primer/topcoat combinations on 7075-T6 Al substrate)
Experimental Control	Unexposed portions and DI water-exposed panels

Specimens	
Acceptance Criteria	Performs as well as unexposed areas of the panel
Reference Document	ASTM D 3359, Methods A & B
Test Equipment	Standard laboratory

Test Results

Table 34 lists the results of the adhesion testing, conducted in accordance with Methods A and B.

Table 34. Adhesion Results

Coating	Substrate	Fluid	Unexposed- Method A	Exposed- Method A	Unexposed- Method B	Exposed- Method B
			5A	5A	5B	5B
_		DI Water	5A	5A	4B	5B
Outer	7075 T-6		5A	5A	4B	5B
Mold Line	Aluminum		5A	5A	4B	5B
Primer		EcoFlo	5A	5A	5B	5B
			5A	5A	5B	5B
			5A	5A	4B	5B
7 36 11		DI Water	5A	5A	4B	5B
Inner Mold Line	7075 T-6		5A	5A	5B	5B
Primer	Aluminum		5A	5A	5B	5B
Primer		EcoFlo	5A	5A	4B	5B
			5A	5A	5B	5B
		DI Water	5A	5A	5B	5B
0.1			5A	5A	5B	5B
Outer	41.40 C41		5A	5A	5B	5B
Mold Line Primer	4140 Steel	EcoFlo	5A	5A	5B	5B
Primer			5A	5A	5B	5B
			5A	5A	5B	5B
			5A	5A	5B	5B
7 34 11		DI Water	5A	5A	5B	5B
Inner Mold Line	4140 Steel		5A	5A	5B	5B
Primer	4140 Steel		5A	5A	5B	5B
Filliel		EcoFlo	5A	5A	5B	5B
			5A	5A	5B	5B
			5A	5A	4B	4B
Outer Mold Line		DI Water	5A	5A	5B	5B
	AZ91E		5A	5A	5B	5B
Primer	Magnesium		5A	5A	5B	5B
1 1111161		EcoFlo	5A	5A	5B	5B
			5A	5A	5B	5B
Inner Mold	AZ91E	DI Water	5A	5A	5B	5B

Line	Magnesium		5A	5A	5B	5B
Primer			5A	5A	5B	5B
			5A	5A	5B	5B
		EcoFlo	5A	5A	5B	5B
			5A	5A	5B	5B
Outer			5A	5A	4B	5B
Mold Line		DI Water	5A	5A	4B	5B
Primer &	7075 T-6		5A	5A	4B	5B
Anti-Static	Aluminum		5A	5A	5B	5B
Rain	7 Hummum	EcoFlo	5A	4A	4B	5B
Erosion Topcoat		Leorio	5A	5A	4B	4B
Inner Mold			5A	5A	4B	5B
Line		DI Water	5A	5A	4B	5B
Primer &	7075 T-6		5A	5A	4B	5B
Anti-Static	Aluminum		5A	5A	4B	4B
Rain	7 11011111111111	EcoFlo	5A	5A	4B	4B
Erosion Topcoat		200110	5A	5A	4B	4B
Outer			5A	5A	0B	0B
Mold Line		DI Water	5A	5A	0B	0B
Primer &	7075 T-6		5A	4A	0B	0B
Rain	Aluminum		5A	5A	0B	2B
Erosion		EcoFlo	5A	4A	0B	3B
Topcoat			5A	5A	0B	3B
Inner Mold			5A	5A	4B	5B
Line		DI Water	5A	5A	4B	5B
Primer &	7075 T-6		5A	5A	4B	5B
Rain	Aluminum		5A	5A	4B	4B
Erosion		EcoFlo	5A	5A	4B	4B
Topcoat			5A	5A	3B	4B

The results of the adhesion testing showed that EcoFlo deicer had no effect on the coating systems. There were no instances where the change in adhesion varied by more than one adhesion scale unit. Poor adhesion was noted for the panels coated with outer mold line primer and rain erosion topcoat, tested for Method B adhesion, but this adhesion issue was consistent with the DI water-exposed samples, as well, indicating a potential panel preparation problem.

4.7.2.4 Pencil Hardness

Test Description

This testing method describes the procedure to perform pencil hardness testing on coated test panels that have been partially exposed to the deicing solution. The first section discusses the immersion procedure, which is the same procedure utilized for the adhesion testing. The second

section describes the pencil hardness test, which was performed on both the exposed and unexposed portions of the test panels.

Immersion - After the paint drying cycle, each test panel was placed in a horizontal position in an oven maintained at $38 \pm 2^{\circ}$ C ($100 \pm 5^{\circ}$ F). The deicing solution was applied to approximately one half the area of each panel and allowed to remain on the panel for 30 minutes. Then, the panels were removed from the oven, rinsed with DI water, and allowed to air dry for 24 hours. Each panel was then visually examined for streaking, discoloration, or blistering of the finish prior to adhesion testing.

<u>Pencil hardness</u> - A coated panel was placed on a firm horizontal surface. The pencil was held firmly against the film at a 45° angle (point away from the operator) and pushed away from the operator in a 6.6-mm (1/4-in.) stroke. The pencil was pushed across the paint film with a firm uniform pressure until a pencil was found that did not cut the film but left a black mark on the surface, whereas the next hardest pencil cut through the film without leaving a black mark. The hardness number of the pencil that cuts the film expressed hardness.

Test Methodology

Parameters	3 x 6 inch coated, deicer-exposed panels were tested at the exposed and unexposed portions of the panels with pencil leads of various hardness to determine the lead that cuts through the coating
Type/Number of Specimens	3 panels per coating system (primer/topcoat combinations on 7075-T6 Al)
Experimental Control Sample	Unexposed portions of test panels plus panels exposed to DI water
Acceptance Criteria	No greater than one pencil hardness difference between the exposed and unexposed portions of the test panels
Reference Document	ASTM D3363
Test Equipment	Standard pencil hardness set

Test Results

Table 35 lists the results of the pencil hardness testing.

Table 35. Pencil Hardness Results

Coating	Fluid	Unexposed	Exposed
		8H	Н
Outer Mold Line	DI Water	8H	2H
Primer & Anti-Static		5H	Н
Rain Erosion Topcoat	EcoFlo	8H	2H
	ECOPIO	8H	4H

T T			
		8H	2H
		3Н	4H
Inner Mold Line	DI Water	3H	5H
Primer & Anti-Static		4H	4H
Rain Erosion Topcoat		4H	2H
Kam Erosion Topcoat	EcoFlo	6H	4H
		6H	5H
		8H	2H
Ot M-111 :	DI Water	4H	F
Outer Mold Line Primer & Rain		8H	Н
Erosion Topcoat	EcoFlo	8H	F
Liosion Topeoat		5H	Н
		8H	Н
		6H	4H
Innan Mald Lina	DI Water	6H	4H
Inner Mold Line Primer & Rain		5H	4H
Erosion Topcoat		5H	4H
Erosion ropcoat	EcoFlo	5H	5H
		8H	4H

The panels painted with inner mold line primer and anti-static rain erosion topcoat, and inner mold line primer with rain erosion topcoat averaged a change in hardness of 1 or 2 units. Some panels were 1 unit or less individually.

The panels painted with outer mold line primer and anti-static rain erosion topcoat, and outer mold line primer with rain erosion topcoat averaged a change in hardness of 5 or 6 units. Some panels had as little as 4 units change in hardness, but still significantly higher than the 1 unit requirement. Again, as seen with the adhesion test panels, there may have been a panel preparation issue related to the outer mold line primer application.

Overall, the panels with the inner mold line primer were less susceptible to the effects of immersion in EcoFlo deicing fluid compared to the panels with the outer mold line primer.

4.8 LO Sealants

4.8.1 LO Sealant Material

One LO sealant material was tested for material compatibility, PR 2200 Class B gap sealant, purchased from PRC DeSoto. The main use of this sealant is to seal and adhere to windshields.

4.8.2 LO Sealant Testing Methods

The LO sealant material was tested for volume swell according to SAE AMS 5127/1. The sealant material was prepared per manufacturer's mixing instructions. The sealant was applied

to polyethylene sheets and after curing, the appropriate sample sizes were cut from the sealant material.

4.8.2.1 Volume Swell

Test Description

The 1 x 3 inch test specimens were cut from the cured sealant. Each specimen was weighed in air (W1) and in water (W2) and then dried. The specimens were then immersed in 900 milliliters (ml) of each deicing material for 7 days at 60°C (140°F) in a closed container. The specimens were then removed from the fluid at the end of the immersion time, dipped momentarily in methanol, and reweighed in air (W3) and water (W4). The percent volume swell was then calculated by the following equation:

Percent Swell =
$$[(W2+W3)-(W1+W4)) / (W1-W2)] * 100$$

<u>Test Methodology</u>

Parameters	1 x 3 inch specimens were cut from cured sealant and exposed to the deicing solutions for 7 days with specimens weighed in air and water both before and after exposure
Type/Number of Specimens	3 sealant specimens
Experimental Control Specimens	3 sealant specimens exposed to DI water
Acceptance Criteria	% volume swell shall be checked for conformance to
	the sealing material specification.
Reference Document	SAE AS 5127/1
Test Equipment	Analytical Balance
	Drying Oven

Test Results

Table 36 lists the results of the volume swell testing for the LO sealant material.

Table 36. Volume Swell for LO Sealant

Sample Name	Sample 1	Sample 2	Sample 3
DI Water Control	-6.22%	-71.7%	-29.3%
EcoFlo-exposed Sample	70.4%	108.9%	209.6%

These results indicate that the sealant material shrunk in size when exposed to DI water and then greatly absorbed the EcoFlo deicer to expand up to more than double in size. Potentially, the material was not fully cured. In addition, peel strength samples peel apart easily, not allowing for an accurate test of peel strength due to severe adhesive failure between the coating system

and the sealant. Given the volume swell results and the adhesive failures of the sealant, no further testing was conducted with the LO sealant material.

4.9 Lubricants and Greases

Various types of lubricants and greases were identified for material compatibility testing with deicing solutions. The purpose of the testing was to determine if the deicing solutions affect the ability of the lubricants and greases to provide wear protection and corrosion protection. Greases were exposed to the deicers by first coating a metal panel with grease, then applying the deicer over the grease, whereas lubricants were mixed with varying concentrations of deicing solutions.

4.9.1 Lubricants and Greases Test Materials

Four greases and four lubricant materials were purchased for testing. The military specification, basic constituent, and supplier of each material are listed in Table 37.

Table 37. Lubricants and Greases

Military Specification	Chemistry	Supplier
MIL-PRF-32014	PAO based grease, containing long chain alkenyl amide borate	Nye Lubricants (374A)
MIL-PRF-81322	PAO based grease	Windward Petroleum (Mobil Grease 28)
MIL-PRF-27617	PFPAE based grease	Du Pont (Krytox 240 AC)
MIL-PRF-83261	Silicone oil based grease	Aerospace Lubricants, Inc. (Tribolube)
MIL-PRF-87257	Lubricant containing synthetic hydrocarbons, adipate ester	Radco Industries (FR257)
MIL-PRF-83282	Lubricant with PAO basestock, adipate ester	Radco Industries (FR282)
MIL-PRF-5606	Lubricant with hydrotreated light napthenic distillate, acrylic copolymer	Radco Industries (FR5606)
MIL-PRF-7808	Aviation engine oil composed of synthetic base stocks	Windward Petroleum (Jet Oil 254)

4.9.2 Lubricants and Greases Testing Methods

Two testing methods were performed to determine the effects of exposure of the EcoFlo deicer to lubricants and greases:

- 1. Humidity testing ASTM D1748
- 2. Torque rheometry AFRL-developed procedure

Torque rheometry testing was only performed on two of the test greases.

4.9.2.1 Humidity Testing

Test Description

Test panels were manufacturer from 1010 Low-Carbon Steel. Dimensions of panels were 2" x 4" x 1/8" with two holes between 1/8" to 3/32" in diameter drilled in the corners along one of the 4" edges. Sampled IDs were indented on one face of the panel. The face opposite of the indented ID was the only surface evaluated for testing. Prior to panel preparation, panels were inspected to ensure they were free of pits, scratches, rust or any other surface imperfections. Panels with imperfections were not tested and replaced. Preparation of the panels for lubricant and grease testing is described in the following paragraphs.

Greases – Panels that were subjected to materials compatibility testing for various greases with deicer were prepared according to the following method. Five 1010 steel panels, measuring 2" x 4" inches were prepared for each testing condition. After visual inspection for imperfections, panels were suspended by use of two hooks made from stainless steel wire through the drilled holes. Panels were immersed in boiling acetone for 5 minutes, then boiling toluene for 5 minutes and allowed to cool to room temperature by hanging undisturbed for a minimum of 15 minutes. After cooling, the panel face that was evaluated and all edges were hand polished with 240 and then 320 grit silicon carbide paper. The hooks were removed from the panel at this time to ensure that the entire test surface was consistently polished. The test face and edges were polished to achieve a clean, active surface for testing. The panel was quickly cleaned with acetone to remove majority of the soot from polishing. Next, the panel was re-suspended with the hooks, and the panel was boiled once again in acetone and toluene for 5 minutes each and cooled to room temperature. At this point the panel was considered clean, and was not handled with hands, especially on the testing face. After cooling, the panel was coated with grease on all sides using a small spatula. A magnet placed in clean plastic baggie was utilized to keep the panel in place without handling it. First the indented ID face and edges were greased (the cleaned test surface shall be against the plastic bag containing the magnet). The wire hooks were used to remove the panel from the magnet and flip the orientation of the panel so the test surface can be greased. Special precautions were taken to ensure that all sides, the face with indented ID and drilled holes were covered with sufficient amounts of grease to prevent any unwanted corrosion. The spatula was then used to remove as much grease as possible from the testing surface, resulting in a very thin layer of grease. (Note: the amount of grease on the edges and face with indented ID is only needed to prevent any unwanted corrosion). The greased panel was then quickly dipped in the deicing fluid, ensuring it was completely immersed, and hung in a dust free environmental for 15 minutes. Any excess material was carefully removed from the bottom of the panel with a lint free towel. Using the hooks, the panel was transferred to a properly labeled rack for testing. Panels that were subjected to the 40°C and 80% relative humidity conditions

were placed in the humidity chamber. Unexposed panels were placed in the laboratory and covered with a hard plastic cover to keep dust and debris from collecting on the test specimens.

Five panels were evaluated per condition. Panels were tested at high temperature (40°C) and high humidity (80%) conditions as well as ambient temperatures in the lab (labeled as unexposed). Control panels tested with the greases only (no deicer contamination) were also run as baselines, along with bare and deicer only panels. Evaluations were taken on each panel after 24 hours of exposure, and daily to note any changes. Final inspection was completed after approximately 168 hours of exposure. Evaluations on panels were completed according to ASTM D610 specifications, which are described in the testing results section.

<u>Lubricants</u> – The panels used for evaluating corrosion of lubricants were prepared in the same fashion as the greases up to contamination of the samples with deicer. For contamination of exposed panels, the test panels were dipped in lubricant mixed with deicing solution at a concentration of 0.25%, 0.5%, and 1.0% deicer. For unexposed samples, panels were dipped in lubricant mixed with deicing solution at a concentration of 1.0% deicer. The test panels were hung in a dust free environment for 15 minutes with any excess fluid removed from the bottom of the panel with a lint free cloth. The suspended panels were transferred to the testing racks. Panels to be subjected to elevated conditions were placed in the chamber. Unexposed panels were placed in the laboratory and covered with a hard plastic cover to keep dust and debris from collecting on the test specimens.

Control panels tested with lubricants only (no deicer) were also prepared and tested as baselines, along with bare and deicer only panels. Evaluations were taken on each panel after 24 hours of exposure, and daily to note if there were any significant changes. Final inspections were completed after approximately 168 hours of exposure. Observations were completed according to ASTM D610 specifications.

Test Methodology

Parameters	1010 steel panels are cleaned, contaminated grease or	
	lubricant with deicer, placed in their respective testing	
	conditions and evaluated for corrosion	
Type/Number of Specimens	Five specimens per grease/deicing solution and	
	lubricant/deicer combination	
Experimental Control Specimen	Bare panels, lubricant/grease baseline panels, and	
	unexposed lubricant and grease panels	
Acceptance Criteria	Comparison corrosion ratings to control and unexposed	
	panels	
Reference Document	CTC-developed procedure, modified ASTM D1748	
Test Equipment	Standard Lab Equipment, Stopwatch, Humidity	
	Chamber	

Test Results

Panels were evaluated according to the specifications in ASTM D610. Distributions of rust can also be classified as spot rusting (S), general rusting (G), pinpoint rusting (P) and hybrid rusting (H). Ratings are denoted with rust grade numeric value followed by a distribution abbreviation (i.e. 9P). Table 38 below gives a brief explanation of the rating system from ASTM D610. For reporting purposes, final evaluations only will be outlined. Additional evaluations are recorded on the laboratory bench sheets.

Table 38. Rust Grade and Description per ASTM D610 Specifications

Rust Grade	Percentage of Area Rusted
10	Less than or equal to 0.01 percent
9	Greater than 0.01 percent and up to 0.03 percent
8	Greater than 0.03 percent and up to 0.1 percent
7	Greater than 0.1 percent and up to 0.3 percent
6	Greater than 0.3 percent and up to 1.0 percent
5	Greater than 1.0 percent and up to 3.0 percent
4	Greater than 3.0 percent and up to 10.0 percent
3	Greater than 10.0 percent and up to 16.0 percent
2	Greater than 16.0 percent and up to 33.0 percent
1	Greater than 33.0 percent and up to 50.0 percent
0	Greater than 50 percent

For testing controls, bare steel panels that were cleaned and polished as well as cleaned panels immersed in deicer only were tested in the elevated humidity and temperature conditions (40°C, 80% RH). Like the lubricants and greases, five panels were tested for each condition. Final evaluations for the "exposed" controls are found below in Table 39.

Table 39. Ratings for Bare and Deicer Only Exposed Panels

Panel ID	Final Evaluation at 168 Hours
	RRA 9P
	RRA 7P
Bare Steel Controls	RRA 8P
	RRA 6P
	RRA 7P
Deicer Only Controls	RRA 9P
	RRA 9P
	RRA 9P
	No Corrosion
	RRA 8P

The bare steel controls had final corrosion ratings ranging from 6-9 with the pinpoint rusting noted. The acronym RRA refers to "red rust observed in the test area." The deicer only control panels had less corrosion than the bare steel after 168 hours of exposure, with ratings at 8-9 and pinpoint rusting. One panel was noted as having no corrosion. When comparing the bare steel and deicer only controls, the deicer appears to act as a barrier against corrosion because ratings gave lower percentages of rusting.

Four greases were evaluated on their compatibility with the deicer solution. Of the four tested, one grease (MIL-PRF-32014) was known to be corrosion resistant, while MIL-PRF-27617 was believed to be corrosion prone. Each grease panel was cleaned, prepared, greased and immersed in deicer according to the method previously described. A control with grease only was exposed to the elevated temperature/humidity conditions as a baseline. Evaluations for grease only baselines are outlined below in Table 40.

Table 40. Ratings for Baseline Grease Only Exposed Panels

Grease Specification	Final Evaluation at 168 Hours
	No Corrosion
MIL-PRF-32014	No Corrosion
(Corrosion Resistant Grease)	No Corrosion
Baseline Panels	No Corrosion
	No Corrosion
	No Corrosion
	No Corrosion
MIL-PRF-81322 Baseline Panels	No Corrosion
Duscinic Funcis	No Corrosion
	No Corrosion
	RRA 4P
MIL-PRF-27617	RRA 3P
(Corrosion Prone Grease)	RRA 5P
Baseline Panels	RRA 5P
	RRA 3P
	RRA 9P
	RRA 9P
MIL-PRF-83261 Baseline Panels	RRA 9P
Dascinic Lancis	RRA 9P
	RRA 9P

The corrosion resistant grease (MIL-PRF-32014) and MIL-PRF-81322 did not show any signs of corrosion after 168 hours of exposure at 40°C and 80% relative humidity. The corrosion prone grease (MIL-PRF-27617) performed the least successful having ratings ranging from 3-5 with pinpoint rust observed. MIL-PRF-83261 also displayed corrosion after 168 hours with ratings of 9. The performance of the baseline grease only panels was used for comparison to the exposed panels that were immersed in deicer.

The four greases were tested at two conditions with the deicer: "humidity" (in the humidity chamber) and "lab conditions" (in laboratory conditions). The comparison between the final exposed and unexposed panels for the greases are outlined below in Tables 41-44.

Table 41. MIL-PRF-32014 Grease with Deicer Final Evaluations

Grease Specification	Final Evaluation After 168 Hours – Humidity	Final Evaluation After 168 Hours – Lab Conditions
	No Corrosion	No Corrosion
MIL-PRF-32014	No Corrosion	No Corrosion
(Corrosion Resistant Grease)	No Corrosion	No Corrosion
with Deicer	No Corrosion	No Corrosion
	No Corrosion	No Corrosion

When comparing the panels exposed to high humidity and high temperature conditions with the lab condition panels, the sets performed the same. No corrosion was observed at either condition during the 168 hours of testing. The evidence of no corrosion after testing also coincides with the MIL-PRF-32014 baseline panels that also did not present any form of corrosion after 168 hours. The MIL-PRF-32014 grease panels performed better than the bare steel control and deicer only controls.

Table 42. MIL-PRF-81322 Grease with Deicer Final Evaluations

Grease Specification	Final Evaluation After 168 Hours – Humidity	Final Evaluation After 168 Hours – Lab Conditions
	No Corrosion	No Corrosion
	No Corrosion	No Corrosion
MIL-PRF-81322 with Deicer	No Corrosion	No Corrosion
	No Corrosion	No Corrosion
	No Corrosion	No Corrosion

When comparing the MIL-PRF-81322 panels exposed to high humidity and high temperature conditions with the lab condition panels, the sets performed the same. No corrosion was observed at either condition during the 168 hours of testing. The evidence of no corrosion after

testing also coincides with the MIL-PRF-81322 baseline panels that also did not present any form of corrosion after 168 hours. The MIL-PRF-81322 grease panels performed better than the bare steel control and deicer only controls.

Table 43. MIL-PRF-27617 Grease with Deicer Final Evaluations

Grease Specification	Final Evaluation After 168 Hours – Humidity	Final Evaluation After 168 Hours – Lab Conditions
	RRA 3P	No Corrosion
MIL-PRF-27617	RRA 4P	No Corrosion
(Corrosion Prone Grease)	RRA 5P	No Corrosion
with Deicer	RRA 4P	No Corrosion
	RRA 3P	No Corrosion

Unlike the two previous greases, the corrosion prone MIL-PRF-27617 grease panel sets did not perform equally at the two testing conditions. Red rust was observed at the test area for the panels subjected to the 40°C and 80% relative humidity conditions yielding rust ratings of 3-5 with pinpoint rusting. The unexposed panels that were subjected to laboratory conditions produced no forms of corrosion after 168 hours of testing. When comparing the humidity-exposed MIL-PRF-27617 with deicer panels to the humidity-exposed baseline panels, the two sets performed similarly. Each set gave ratings of 3-5 with pinpoint rust. The corrosion prone grease resulted in an identical range of rust ratings for both sets. Regardless if the panel was immersed in deicer prior to testing or not, high humidity and high temperature conditions resulted in the similar results for this grease. The humidity-exposed conditions with the corrosion prone grease (MIL-PRF-27617) resulted in corrosion on the panels after 168 hours. With rusting rates between 3 and 5, the humidity-exposed MIL-PRF-27617 grease panels yielded higher percentages of corrosion after 168 hours than the bare steel controls and deicer only controls.

Table 44. MIL-PRF-83261 Grease with Deicer Final Evaluations

Grease Specification	Final Evaluation After 168 Hours – Humidity	Final Evaluation After 168 Hours – Lab Conditions
	RRA 1P	No Corrosion
	RRA 3P	No Corrosion
MIL-PRF-83261 with Deicer	RRA 2P	No Corrosion
	RRA 3P	No Corrosion
	RRA 5P	No Corrosion

The MIL-PRF-83261 grease sets did not perform equally for the two testing conditions. Red rust was observed at the test areas for the panels subjected to the 40°C and 80% relative humidity

conditions. Evaluations at 168 hours gave rust ratings of 1-5 with pinpoint rusting. The panels that were subjected to laboratory conditions yielded no forms of corrosion after 168 hours of testing. The deicer exposed MIL-PRF-83261 panels had more corrosion observed after exposure than the baseline and unexposed panels. The MIL-PRF-83261 deicer exposed panels also had more corrosion than the bare steel (rust ratings of 6-9) and deicer only controls (rust ratings of 8-9). Because humidity-exposed deicer/grease panels performed the least successful of any conditions (even considering the baseline and controls), the deicer and MIL-PRF-83261 grease combination appears to affect the corrosion resistance performance of the grease. Contamination or contact with the deicer increases the percentage of corrosion observed after 168 hours at 40°C and 80% relative humidity.

Four lubricants were evaluated on their compatibility with the deicer solution. Lubricants were contaminated with various concentrations of deicer in the solution (0.25%, 0.5% and 1.0%). Each lubricant panel was cleaned, prepared, greased and immersed in lubricant/deicer solutions according to the method previously described. A control with the lubricant only was exposed to the elevated temperature/humidity conditions as a baseline. Final evaluations for lubricant baselines are outlined below in Table 45.

Table 45. Ratings for Baseline Lubricant Only Exposed Panels

Lubricant Specification	Final Evaluation at 168 Hours
	RRA 5P
	RRA 6P
MIL-PRF-87257 Baseline Panels	RRA 8P
Duseline Tuneis	RRA 6P
	RRA 8P
	RRA 5P
	RRA 4P
MIL-PRF-83282 Baseline Panels	RRA 6P
Buseline Tuners	RRA 8P
	RRA 9P
	Thin Flash Rust Color Approximately 50%
MIL-PRF-5606	RRA 9P
Baseline Panels	RRA 9P
	RRA 8P
	RRA 8P
	RRA 9P
MIL-PRF-7808 Baseline Panels	RRA 8P
Baseline Fancis	RRA 9P

RRA 6P
RRA 5P

The baseline lubricant panels were exposed to high humidity (80%) and elevated temperature (40°C) for 168 hours before final evaluations were taken. Corrosion was observed on each of the four lubricants tested. MIL-PRF-87257 yielded red rust corrosion in the test area with a rust rating range of 5-8 with pinpoint rusting. Lubricant MIL-PRF-83282 also gave pinpoint red rusting on all baseline panels with a range of 4-9. MIL-PRF-5606 gave red rusting corrosion on its panels ranging in 8-9 with one panel having flash rusting observed on approximately 50% of the testing area. The final lubricant, MIL-PRF-7808, also gave red pinpoint rusting. The rusting rating range for these panels was 5-9. The performance of the baseline lubricant only panels will be used for comparison of the exposed panels that were contaminated with various concentrations of deicer.

Each lubricant was tested at two testing conditions: either humidity-exposed (40°C and 80% relative humidity in the humidity chamber) or unexposed laboratory conditions. To begin, lab conditions panels were only tested with 1.0% deicer contamination to test "worst case" scenario to determine if deicer would affect the corrosion performance of the lubricants at ambient conditions. Results from the 1.0% deicer in lubricants for unexposed panels are outlined below in Table 46.

Table 46. Ratings for Lubricant with 1.0% Deicer Unexposed Panels

Lubricant Specification	Final Evaluation at 168 Hours – Lab Conditions	
	No Corrosion	
	No Corrosion	
MIL-PRF-87257 with 1.0% Deicer	No Corrosion	
with 1.070 Beleef	No Corrosion	
	No Corrosion	
	No Corrosion	
	No Corrosion	
MIL-PRF-83282 with 1.0% Deicer	No Corrosion	
with 1.0% Delect	No Corrosion	
	No Corrosion	
MIL-PRF-5606 with 1.0% Deicer	No Corrosion	
	No Corrosion	

MIL-PRF-7808 with 1.0% Deicer	No Corrosion
	No Corrosion

Panels immersed in lubricant with 1.0% deicer solutions were exposed to ambient laboratory conditions before receiving the final evaluation. All lubricant/deicer panels performed the same. No corrosion was observed after 168 hours. Because no corrosion was formed at the 1.0% deicer concentration, the 0.25% and 0.5% deicer lubricant panels were not tested in lab conditions.

For the humidity-exposed conditions, the four lubricants were tested at three concentrations of deicer in the solution: 0.25%, 0.5% and 1.0%. These panels were exposed to the humidity chamber (set at parameters 40°C and 80% relative humidity) with final evaluations being completed after approximately 168 hours. The comparisons between the humidity-exposed panels at various deicer concentrations are outlined below in Tables 47-50.

Table 47. MIL-PRF-87257 Lubricant with Deicer Final Evaluations

	Final Evaluation After 168 Hours – Humidity-exposed			
Lubricant Specification	With 0.25% Deicer	With 0.5% Deicer	With 1.0% Deicer	
	RRA 7P	RRA 7P	RRA 5P	
MIL-PRF-87257	RRA 9P + Flash Rust	RRA 6P	RRA 4P	
	RRA 8P	RRA 8P	RRA 6P	
	RRA 9P + Flash Rust	RRA 9P	RRA 6P	
	RRA 8P	RRA 6G	RRA 8P	

Lubricant MIL-PRF-87257 was tested with 0.25%, 0.5% and 1.0% deicer contamination in the lubricant. The final evaluations taken at 168 hours were outlined above. Panels with 0.25% deicer yielded red pinpoint rusting and had ratings ranging between 7 and 9. Panels with 0.5% deicer yielded similar red pinpoint rusting (one panel had general rusting) with ratings between 6 and 9. Finally the 1.0% deicer solutions gave red rusting on the test area with ratings of pinpoint rusting ranging 4-8. In general the higher the concentrations of deicer, larger percentages of corrosion were present on the panel test surface. When comparing to the MIL-PRF-87257 baseline, the 0.25% deicer solution had lower percentages of corrosion on average than the baseline. The 0.5% deicer solution performed as well as the baseline, while the 1.0% deicer had larger percentages of corrosion. Regardless of deicer concentration when comparing the 1.0% deicer panels, the high humidity and elevated temperature conditions affect the corrosion protection performance of the lubricant after 168 hours of testing.

Table 48. MIL-PRF-83282 Lubricant with Deicer Final Evaluations

	Final Evaluation After 168 Hours – Humidity-exposed			
Lubricant Specification	With 0.75% Deicer V		With 1.0% Deicer	
	RRA 8P	RRA 9P	RRA 5P	
MIL-PRF-83282	RRA 8P	RRA 7G	RRA 6P	
	RRA 7P	RRA 7P	RRA 8P	
	RRA 8P	RRA 6P	RRA 6P	
	RRA 7P	RRA 5P	RRA 8P	

Lubricant MIL-PRF-83282 was tested with 0.25%, 0.5% and 1.0% deicer contamination in lubricants. The final evaluations were taken at 168 hours. Panels with 0.25% deicer yielded red pinpoint rusting and had ratings ranging 7-8. Panels with 0.5% deicer yielded similar red pinpoint rusting (one panel had general rusting) with ratings between 5 and 9. Finally the 1.0% deicer solutions gave red rusting on the test area with ratings of pinpoint rusting ranging 5-8. In general the higher the concentrations of deicer, the larger the percentage of corrosion on the panel test surface. When comparing to the MIL-PRF-83282 baseline, the 0.25% deicer solution had lower percentages of corrosion overall than the baseline. The 0.5% and 1.0% deicer solution performed as well as the lubricant baseline as an overall trend.

Table 49. MIL-PRF-5606 Lubricant with Deicer Final Evaluations

	Final Evaluation After 168 Hours – Humidity-exposed			
Lubricant Specification	With 0.25% Deicer	With 0.5% Deicer	With 1.0% Deicer	
	RRAP 8P	RRA 5P	RRA 6P	
	RRA 6P	RRA 8P	RRA 9P	
MIL-PRF-5606	RRA 5P	RRA 7P	RRA 5P	
	RRA 6P	RRA 5P	RRA 4P	
	RRA 6P + Flash Rust	RRA 7P	RRA 5P	

Lubricant MIL-PRF-5606 was tested with 0.25%, 0.5% and 1.0% deicer contamination in the lubricant. The panels were also placed in the humidity chamber for testing and final evaluations taken at 168 hours. Panels with 0.25% deicer yielded red pinpoint rusting and had ratings ranging 5 to 8. Panels with 0.5% deicer yielded similar red pinpoint rusting with ratings between 5 and 8. Finally the 1.0% deicer solutions gave red rusting on the test area with ratings of pinpoint rusting ranging 4 to 9. There was no obvious trend associated with the rusting ratings and deicer concentrations. When comparing all deicer/lubricant panel performance to the MIL-PRF-5606 baseline corrosion ratings, no deicer sets performed as well as the baseline. The deicer/MIL-PRF-5606 sets also did not perform as well as the bare steel or deicer only controls.

The deicer and MIL-PRF-5606 combination appears to affect the corrosion resistance performance when subjected to elevated humidity and temperature conditions.

Table 50. MIL-PRF-7808 Lubricant with Deicer Final Evaluations

	Final Evaluation After 168 Hours – Humidity-exposed			
Lubricant Specification With 0.25% Deicer		With 0.5% Deicer	With 1.0% Deicer	
	RRA 6P	RRA 4P	RRA 7P	
MIL-PRF-7808	RRA 4P	RRA 8P	RRA 3P	
	RRA 5P	RRA 6P	RRA 8P	
	RRA 5P	RRA 4P	RRA 5P	
	RRA 4P	RRA 4P	RRA 6P	

Lubricant MIL-PRF-7808 was tested with 0.25%, 0.5% and 1.0% deicer contamination in the lubricant. The final evaluations taken at 168 hours were outlined in the table. Panels with 0.25% deicer yielded red pinpoint rusting and had ratings ranging 4 to 6. Panels with 0.5% deicer yielded similar red pinpoint rusting with ratings between 4 and 8. Finally the 1.0% deicer solutions gave red rusting on the test area with ratings of pinpoint rusting ranging 3 to 8. There was no obvious trend associated with the rusting ratings and deicer concentrations. When comparing all deicer/lubricant panel performance to the MIL-PRF-7808 baseline corrosion ratings, no humidity-exposed deicer panel sets performed as well as the baseline. The deicer/MIL-PRF-5606 sets also did not perform as well as the bare steel or deicer only controls. The deicer and MIL-PRF-7808 combination appears to affect the corrosion resistance performance when subjected to elevated humidity and temperature conditions.

Overall when 1.0% deicer was added to the lubricants, panels had no observed corrosion after 168 hours at ambient laboratory conditions. When panels prepared with similar deicer concentration were subjected to 40°C and 80% relative humidity, corrosion was observed at various percentages.

4.9.2.2 Torque Rheometry

Test Description

This test method describes how the rheology characteristics of greases was determined using a torque rheometer. This testing was conducted with the use of an environmental test chamber set at -54° C (-65°F). Two, one-inch aluminum plates were arranged one millimeter apart, with deicing solution-exposed grease between the plates. The top plate was rotated at 1 rpm while the lower plate remained immobile. The initial torque was recorded and then the test was run for 20 minutes at -54° C (-65°F). The torque value after 20 minutes was recorded as the running torque.

Test Methodology

Parameters	2, 1-inch aluminum plates with deicer-exposed grease in between that was cooled to -54°C (-65°F), with one plate turned at 1 rpm for 20 minutes
Type/Number of Specimens	Three tests per grease/deicing solution combination
Experimental Control Specimen	Test unexposed grease
Acceptance Criteria	Compare torque measurements to unexposed samples
Reference Document	AFRL-developed procedure
Test Equipment	Torque Rheometer
	Environmental Test Chamber

Test Results

Table 51 shows the results of change in viscosity and torque over the 20 minute exposure time, as tested by ATS Rheosystems. Only the MIL-PRF-32014 and MIL-PRF-83261 greases were tested. The report from ATS Rheosystems is attached as Appendix A.

Table 51. Torque Rheometry Results for Greases

Sample Name	Viscosity (Pa's)		Torqu	e (Nm)
	0 minutes	20 minutes	0 minutes	20 minutes
MIL-PRF-32014	47660	19770	0.03366	0.01372
MIL-PRF-32014	32510	14460	0.02263	0.00998
+ EcoFlo				
MIL-PRF-83261	1301	389.3	0.000906	0.00027
MIL-PRF-83261	1117	698.7	0.000774	0.000485
+ EcoFlo				

The two greases tested showed different trends when tested with the additional of the EcoFlo deicer. The MIL-PRF-32014 grease had decreases in both viscosity and torque with the addition of deicer, both initially, and over the 20 minute run time. However, the MIL-PRF-83261 grease actually had less of a change in viscosity and torque over the test time with the addition of deicer.

4.10 Cannon Electrical Plug Connectors

Cannon electrical plug connectors are used throughout aircraft and deicing trucks to form electrical connections for a number of utilities and critical components. It is of key importance to determine the effects of deicing materials on these connectors, especially related to electrical resistance, conductivity, and insulation integrity.

4.10.1 Cannon Electrical Plug Connector Test Materials

The specific type of connectors that were used for testing was MIL-STL-38999 Series III subminiature cylindrical type connectors. Three straight plugs, three boxed receptacles, and three mated plug/receptacle pairs were evaluated.

4.10.2 Cannon Electrical Plug Connector Test Methods

Three testing methods and an immersion procedure were used to evaluate the effects of the deicing materials on the connectors. These procedures were:

- 1. Fluid Immersion Test Procedure for Electrical Connectors Energy Information Administration (EIA)-364-10C
- 2. Withstanding Voltage Test Procedure for Electrical connectors, Sockets and Coaxial Contacts EIA-364-20C
- 3. Insulation Resistance Test Procedure for Electrical Connectors, Sockets, and Coaxial Contacts EIA-364-21C
- 4. Shell to Shell and Shell to Bulkhead Resistance Test Procedure for Electrical Connectors EIA-364-83

Pre-immersion testing included insulation resistance and shell-to-shell conductivity (mated pairs). After immersion, the connectors were tested for insulation resistance, shell-to-shell conductivity (mated pairs), and dielectric voltage withstand.

4.10.2.1 Fluid Immersion

Test Description

The mated and unmated connectors were exposed to a deicing solution heated to 90°C (194°F) for 5 minutes. After immersion, the connectors were air dried for one hour and optically inspected for degradation. This immersion cycle was then repeated 7 times. The immersion cycles for the mated pairs were similar to the unmated pairs, but the specimens were oven-cured for 6 hours instead of one hour. In addition, the amount of torque that was initially required to take apart the mated pairs was recorded (see method EIA-364-13) before immersion.

<u>Test Methodology</u>

Parameters	Plugs, receptacles, and mated pairs were exposed to deicers heated to 90°C (194°F) for 5 minutes
Type/Number of Specimens	3 plugs, 3 receptacles, and 3 mated pairs
Experimental Control Specimen	N/A
Acceptance Criteria	N/A
Reference Document	EIA-364-10C (formerly MIL-STD-1344A Method
	1016)
Test Equipment	General labware
	Torque wrench/transducer

Test Results

The immersion testing was performed for the unmated pairs only. Testing of the mated pairs is being conducted and the results will be reported at a later date, with this report being amended when the test results are available.

4.10.2.2 Insulation Resistance

Test Description

The purpose of this test was to determine the resistance offered by the insulation materials and the various seals of a connector to produce a leakage of current. Insulation resistance was measured between the adjacent pin positions and from pins to shell for both mated and unmated connectors. The connecters were tested using the HIPOT tester at a voltage of 500 VAC for a two minute time period. The testing was conducted pre-immersion and post-immersion.

Test Methodology

Parameters	Mated and unmated connectors were tested with a HIPOT tester set at 500 VAC for 2 minutes to determine if current leaks existed between adjacent pins and from pins to shell
Type/Number of Specimens	3 each mated and unmated pairs exposed to deicing solution per immersion method
Experimental Control Specimen	3 each mated and unmated pair prior to deicing solution immersion
Acceptance Criteria	Compare to pre-immersion results
Reference Document	EIA-364-21C (formerly MIL-STD-1344A Method
	3003.1)
Test Equipment	HIPOT Tester

Test Results

Table 52 lists the results of the insulation resistance test. Each measurement position is identified as pin-to-pin or pin-to-shell. A pass rating means that the current did not leak during the two-minute test period. A fail rating indicates that the 500 VAC could not be maintained and there was a failed connection due to damage or contamination between the connection points.

Table 52. Insulation Resistance Test Results

Sample	Connection	Pre-immersion		Post-in	mersion
Name		Plug	Receptacle	Plug	Receptacle
Unmated	Pins B to C	Pass	Pass	Pass	Pass
Pair - 1	Pins B to D	Pass	Pass	Pass	Pass
	Pins C to D	Pass	Pass	Pass	Pass
	Pin B to Shell	Pass	Pass	Pass	Pass
	Pin C to Shell	Pass	Pass	Pass	Pass
	Pin D to Shell	Pass	Pass	Pass	Pass
Unmated	Pins B to C	Pass	Pass	Pass	Pass
Pair - 2	Pins B to D	Pass	Pass	Pass	Pass

	Pins C to D	Pass	Pass	Pass	Pass
	Pin B to Shell	Pass	Pass	Pass	Pass
	Pin C to Shell	Pass	Pass	Fail	Pass
	Pin D to Shell	Pass	Pass	Fail	Pass
Unmated	Pins B to C	Pass	Pass	Fail	Pass
Pair - 3	Pins B to D	Pass	Pass	Fail	Pass
	Pins C to D	Pass	Pass	Fail	Pass
	Pin B to Shell	Pass	Pass	Fail	Pass
	Pin C to Shell	Pass	Pass	Fail	Pass
	Pin D to Shell	Pass	Pass	Fail	Pass
Mated	Pins B to C	Pass	NA		
Pair - 1	Pins B to D	Pass	NA		
	Pins C to D	Pass	NA		
	Pin B to Shell	Pass	NA		
	Pin C to Shell	Pass	NA		
	Pin D to Shell	Pass	NA		
Mated	Pins B to C	Pass	NA		
Pair - 2	Pins B to D	Pass	NA		
	Pins C to D	Pass	NA		
	Pin B to Shell	Pass	NA		
	Pin C to Shell	Pass	NA		
	Pin D to Shell	Pass	NA		
Mated	Pins B to C	Pass	NA		
Pair - 3	Pins B to D	Pass	NA		
	Pins C to D	Pass	NA		
	Pin B to Shell	Pass	NA		
	Pin C to Shell	Pass	NA		
	Pin D to Shell	Pass	NA		

Again, once testing is complete for the mated pairs, this report will be amended to add the test results. Results for the unmated pairs show all passing ratings for pre-immersion measurements from pin-to-pin and pin-to-shell. Some failures were noted after immersion due to contamination or conductivity of the deicing solution. From past testing programs, any failures due to deicer exposure occur for the unmated pairs, not the mated pairs.

4.10.2.3 Shell-to-Shell Conductivity

Test Description

The purpose of this test was to determine the electrical conduction of the connector shell under simulated service conditions. A test current of 1.0 ± 0.1 ampere at 1.5 volts direct current (VDC) maximum was passed through the shells of the mated connectors. A voltmeter and an ammeter were used to measure the voltage drop across the mated connector from the accessory thread on the plug to the mounting flange on the receptacle. For the square flange receptacles, the

measurements were taken next to the mounting hole. For single hole mount receptacles, the measurements were taken adjacent to the o-ring from the front or mounting side of the flange.

Test Methodology

Parameters	Mated pairs had a test current applied to the shells, with measurements taken from the accessory thread of
	the plug to the mounting flange of the receptacle to determine voltage drop
Type/Number of Specimens	3 mated pairs exposed according to immersion method
Experimental Control Specimen	3 mated pairs prior to immersion
Acceptance Criteria	< 3.0 milivolts for new connectors; compare to pre-
	immersion results
Reference Document	EIA-364-83 (formerly MIL-STD-1344A Method 3007)
Test Equipment	Multimeter
	HIPOT Tester or other power supply

Test Results

Results will be updated once testing is complete. Initial readings for the mated pairs were passing values of < 3.0 mV for all three assembled connectors.

4.10.2.4 Dielectric Voltage Withstand Test

Test Description

The purpose of performing the dielectric withstanding voltage test was to prove that a given electrical connector can operate safely at its rated voltage. The dielectric withstanding voltage was established as 75% of the minimum breakdown voltage of the connector, with the test connectors rated to 1800 VAC. The test voltage for the connectors was applied between the most closely spaced contacts and between connector shell and the closest contact to the shell. The test voltage was raised from zero to the specified value of 1800 VAC as uniformly as possible, at a rate of approximately 500 V/s. The voltage was maintained at 1800 V for 1 minute. The fault indicator was monitored for evidence of disruptive discharge and leakage current.

<u>Test Methodology</u>

Parameters	Mated and unmated pairs were tested with a HIPOT
	tester to 1800 V and maintained for 1 minute, checking
	for current leakage or disruption
Type/Number of Specimens	3 each mated and unmated pairs exposed to deicing
	solution per the immersion method
Experimental Control Specimens	N/A
Acceptance Criteria	75% of 1800 VAC and visual inspection of mated

	connector's ability to seal connection
Reference Document	EIA-364-20C (formerly MIL-STD-1344A, Method
	3001.1)
Test Equipment	Multimeter
	HIPOT Tester

Test Results

Table 53 lists the results of the voltage withstand test for the unmated pairs. This table will be updated with the test results for the mated pairs once testing is complete.

Table 53. Voltage Withstand Test Results

Sample	Connection	Post-imme	ersion
Name		Plug	Receptacle
Unmated	Pins B to C	Pass	Pass
Pair - 1	Pins B to D	Pass	Pass
	Pins C to D	Pass	Pass
	Pin B to Shell	Pass	Pass
	Pin C to Shell	Pass	Pass
	Pin D to Shell	Pass	Pass
Unmated	Pins B to C	Pass	Pass
Pair - 2	Pins B to D	Pass	Pass
	Pins C to D	Pass	Pass
	Pin B to Shell	Pass	Pass
	Pin C to Shell	3.83 mA/1.8 sec	Pass
	Pin D to Shell	Pass	Pass
Unmated	Pins B to C	3.28 mA/0.1 sec	Pass
Pair - 3	Pins B to D	3.18 mA/3.8 sec	Pass
	Pins C to D	3.79 mA/2.6 sec	Pass
	Pin B to Shell	4.26 mA/0.9 sec	Pass
	Pin C to Shell	4.62 mA/0.1 sec	Pass
	Pin D to Shell	4.68 mA/1.5 sec	Pass
Mated	Pins B to C		
Pair - 1	Pins B to D		
	Pins C to D		
	Pin B to Shell		
	Pin C to Shell		
	Pin D to Shell		
Mated	Pins B to C		
Pair - 2	Pins B to D		
	Pins C to D		

	Pin B to Shell	
	Pin C to Shell	
	Pin D to Shell	
Mated	Pins B to C	
Pair - 3	Pins B to D	
	Pins C to D	
	Pin B to Shell	
	Pin C to Shell	
	Pin D to Shell	

Test results for the unmated pairs shows that most of the plugs that failed insulation resistance also showed a current leakage, also a failure, for voltage withstand. Again, contamination or conductivity of the deicer are the most likely reasons for the faults.

4.11 High Velocity Oxygen Fuel (HVOF) Coating

HVOF coatings are being used as replacements for electroplated hard chromium coatings on aircraft landing gear and actuator components, as well as numerous overhaul and rework operations at maintenance facilities. The purpose of this testing was to determine if the deicing materials have any corrosive effects on the HVOF coating.

4.11.1 HVOF Coating Test Material

To conduct this testing, 4340 steel panels, 2 x 2-inch dimension, were purchased and coated with 83% Tungsten Carbide (WC)-17% Cobalt (Co) HVOF coating by a reputable vendor. White Engineering is applying the HVOF coating and they have not returned the test panels for testing at the issuance of this report. Once the panels are received, testing will be conducted and this report will be amended to include the results.

4.11.2 HVOF Testing Methods

Two testing methods were employed to determine the corrosive effects of deicing materials on the HVOF coating:

- 1. Alternate Immersion Testing ASTM G-31
- 2. Humidity Testing ASTM D 1748

4.11.2.1 Alternate Immersion

Test Description

Three HVOF-coated specimens were measured for surface roughness, wiped clean with MEK and then weighed to the nearest mg on an analytical balance. The specimens were placed in the SCC chamber, which was programmed with the following parameters: 1) submerge specimens in the deicing fluid for 10 minutes, 2) drain and then air dry specimens for 50 minutes, and 3) repeat steps one and two continuously for 20 days. The specimens were checked for corrosion

daily. After removal from the chamber, the specimens were weighed and examined for staining, pitting, exfoliation, and corrosion product buildup. Also, all specimens were digitally photographed and again tested for surface roughness.

Test Methodology

Parameters	HVOF-coated specimens exposed to deicing solution	
	for a 20-day cycle of 10 minutes submerged in deicing	
	solution and 50 minutes air dry, with surface roughness	
	measurements before and after test	
Type/Number of Specimens	3 specimens	
Experimental Control Specimens	None	
Acceptance Criteria	No corrosion present or staining, pitting, exfoliation	
	due to corrosion.	
Reference Document	ASTM G-31	
Test Equipment	Stress Corrosion Cracking Chamber	
	Analytical Balance	

Test Results

This report will be amended at a later date to include testing results for this test method. Samples have not been received by the vendor.

4.11.2.2 Humidity Testing

Test Description

Specimens were cleaned with MEK and then weighed to the nearest mg on an analytical balance. Deicing solution was applied by dipping the specimens into the deicing fluid for one minute. The specimens were then drained and suspended in a humidity cabinet at 38°C (100°F) and 95% relative humidity for 20 days. Deicing solution was reapplied after 24 hours, 48 hours, 72 hours 96 hours and then once weekly until the end of the test run. After removal from the chamber, the specimens were weighed and evaluated for rust dots by size and amount on the surface of each rod. Lastly, all specimens were digitally photographed and again tested for surface roughness.

<u>Test Methodology</u>

Parameters	HVOF-coated specimens immersed in deicing solution for 1 minute then placed in a humidity cabinet for 20 days, with deicing solution reapplied after 24, 48, 72, and 96 hours, then weekly until test conclusion, with surface roughness measurements before and after test
Type/Number of Specimens	3 specimens
Experimental Control Specimen	N/A
Acceptance Criteria	Less than 3 rust dots smaller than 1mm in diameter
Reference Document	Modified ASTM D1748-02

Test Equipment	Humidity/Corrosion Chamber
	Analytical Balance
	Stereomicroscope

Test Results

This report will be amended at a later date to include testing results for this test method. Samples have not been received by the vendor.

4.12 Plastic Windows

Plastic materials serve many purposes on aircraft and ground support vehicles. These materials can be clear or opaque, in order to serve truly as a window/hatch or as a support structure. The materials tested in this method were purchased as transparent.

4.12.1 Plastic Windows Testing Material

Substrate materials identified for this testing section include military specification plastics as listed in Table 54.

Table 54. Plastic Window Materials for Evaluation

Military Specification	Description	Stress Level	Vendor
MIL-P-5425	Poly II cast acrylic sheet, transparent, heat resistant	Outer fiber stress of 3000 psi	Spartech Polycast

4.12.2 Plastic Windows Testing Method – Crazing Effect

This test method was used to determine the crazing effect of deicing fluid on acrylic plastic under stress. The crazing effect is described as clouding or etching of the plastic material, causing the plastic to weaken at the point of stress.

Test Description

Six test specimens were cut from each of the three plastic materials listed in Table 12, with the dimensions: 1 x 7 x 0.25 inches and edges that were a smooth machined surface. Test specimens were conditioned before testing, at 24 ± 3 °C (75 ± 10 °F) and 50 ± 5 % relative humidity for a minimum of 24 hours.

The test specimens were then loaded into a cantilever beam system as described in ASTM F484, and stressed according to the levels listed in Table 12 for 10 minutes. After the stress period, the test specimens were visually inspected to ensure that no crazing has been initiated. If the test specimens pass inspection, then they were again loaded into the cantilever beam for further testing as follows. With the test specimen loaded, a ½ to 5/8-inch square absorbent cotton or

flannel swatch was placed directly on the center of the tension surface of the test specimen (directly over the fulcrum). Once in place, the cotton swatch was completely soaked with the deicing fluid, and kept moist for the duration of the 8 hour test. The test specimens were inspected for crazing or degradation at the following time intervals: 30 minutes, 1 hour, 2 hours, 4 hours, and 8 hours. To inspect the test specimen, the moist swatch was moved approximately 2 inches toward the loaded end, and the area was wiped clean using a cloth wet with DI or distilled water. The surface of the test specimen was visually inspected for evidence of crazing, cracks, or etching. Crazing that has initiated at the edge of the specimen was disregarded unless it grew and extended across the specimen. If at any point during the test the specimens show evidence of crazing, cracking, or etching, the test was terminated (even if the 8 hour duration has not been reached). Reporting included: the deicing fluid tested, the type of plastic material tested, description of the specimen surface after testing, and the duration of exposure before test failure (if applicable).

Test Methodology

Parameters	Specimens were stressed to specified level in the cantilever beam, exposed to deicing fluid for a duration of 8 hours, and periodically checked for crazing	
Type/Number of Specimens		
	6 specimens	
Experimental Control Specimens	2 non-stressed, unexposed specimens	
Acceptance Criteria	No crazing, cracking, etching, or staining of the test	
	specimen	
Reference Document	ASTM F 484, ADS-61A-PRF	
Test Equipment	Cantilever Beam; Concentrated Columnated Light	
	Source	

Results and Discussion

Table 55 lists the results of the crazing effect study for the cast acrylic sheet material.

Table 55. Crazing Effects Results for Plastic Window Material(s)

Deicer	Plastic Material	Sample Description	Results
	Poly II Cast Acrylic Sheet	Control 1	N/A -
		Control 2	Unexposed/unstressed controls
EcoFlo		Sample 1	
		Sample 2	
		Sample 3	No cracking or
		Sample 4	crazing after 8 hrs
		Sample 5	
		Sample 6	

N/A – Not applicable; utilized for visual comparisons to test samples to evaluate affects of the deicer on samples

The deicing solution had no impact on the poly II cast acrylic materials based on the reported results of no signs of cracking or crazing when evaluated after being stressed and exposed to the deicing solution for 8 hours.

5.0 CONCLUSION

The results of the materials compatibility testing of the EcoFlo deicer, manufactured by Octagon Process, Inc, show that this deicer had comparable results to the baseline Type I deicer, Octaflo EF, also manufactured by Octagon Process, Inc. The EcoFlo formulation utilizes less PG, substituting glycerin. Glycerin is relatively inert, chemically, with regard to effect on materials.

To summarize the results of each material category, Table 56 presents overall pass/fail, as compared to the acceptance criteria.

Table 56. Summary of Materials Compatibility Testing

Material Category	Test Method	Result
Metallic Materials	Alternate Immersion	Pass
	Stress Corrosion Cracking	Pass
	Total Immersion Corrosion	Pass
	Effect on Unpainted	Pass
	Surfaces	
PMC Material	In-plane Shear	Pass
	Barcol Hardness	Pass
	Glass Transition Temp	Inconclusive
	Sandwich Corrosion	Pass
	Thermal Oxidative Stability	Pass
	Percent Weight Gain	Pass
Elastomeric Materials	UTS/Percent Elongation	Pass
	100% and 300% Modulus	Pass
	Peel Strength/% Cohesive	Pass
	Failure	
	Shore A Hardness	Pass
	Percent Volume Swell	Fail
Aircraft Wire Insulation	Immersion/Bend	Pass
	Voltage Withstand	Pass
Carbon-carbon Brake	Oxidation Resistance	Comparable to control
Infrared Windows	Change in transmission	Pass
LO Coatings	Liquid Uptake	Pass
	Adhesion	Pass – issue with outer mold line primer
	Pencil Hardness	Pass – issue with outer mold line primer
LO Sealant	Volume swell	Fail – potential cure issue
Lubricants and greases	Humidity	Pass
	Torque Rheometry	Pass
Cannon Plugs	Insulation Resistance	Unmated only – some failures
	Shell-to-shell conductivity	Results Pending
	Voltage Withstand Testing	Unmated only – some failures
HVOF	Humidity Testing	Results Pending

	Alternate Immersion	Results Pending
Plastic Windows	Crazing Effect	Pass



APPENDIX A



Monday, November 1, 2010

Leanne Debias Concurrent Technologies Corporation 100 CTC Drive Johnstown, PA, 15904

RE: Report August 18, 2008

Dear Leanne,

We have finished the rheological tests on the four (4) grease samples you supplied. All the tests have been conducted on a NOVA Rheometer equipped with our RheoPolymer LN_2 Temperature Cell. The upper limit of torque of this particular NOVA Rheometer is 2.0×10^{-1} Nm. The primary goal of this project is to obtain the viscosity and torque after constant rotation at a speed of 1 RPM for varied grease samples. The testing geometry was a parallel plate with diameter of the upper plate of 15.0 mm and lower plate of 30.0 mm, and the gap separation of 1.000 mm. Both plates are roughened to avoid slippage between the sample and the plates. For such setup, the desired rotation speed of 1 RPM yields a shear rate of 0.785 s⁻¹. The temperature was set to be constant at $-54^{\circ}C$.

Data Summary:

The values of viscosity and torque obtained for all the samples at 0 and 20 minutes were averaged over a 30 seconds window and are summarized in Table I. Comparison of these results indicates that different types of anti-icer have different effects on the grease samples.

REOLOGICA Instruments Guarantee

The results generated in this report by this model of rheometer can be routinely reproduced when used under the same laboratory conditions anywhere in the world.

Table I: Shear viscosity and torque for all samples at 0 and 20 minutes of constant shear rate experiment. Transient condition SAOS complex viscosity is shown for "27617".

	Viscosity (Pa*s) 0 minute 20 minutes		Torque (Nm)	
			0 minute	20 minutes
32014	4.766E+4	1.977E+4	3.366E-2	1.372E-2
32014+Type I	3.251E+4 1.446E+4		2.263E-2	9.98E-3
83261	1.301E+3	3.893E+2	9.063E-4	2.703E-4
83261+Type I	1.117E+3	6.987E+2	7.744E-4	4.848E-4

Sample Preparation and Experimental Setup:

The grease sample was loaded using a spatula to the lower plate, which had been pre-zeroed at -54° C. The loading normal force was set and controlled by the rheometer's patented Differential Pressure Normal Force sensor at 10 N while the 15.0 mm diameter upper plate was lowered to 0.100 mm above the target gap. The excess sample was trimmed and the plate was lowered to the run gap of 1.000 mm. Finally the sample was allowed to rest for 360 seconds for both stress and thermal equilibrium. For the two samples with deicer added, the sample was mixed vigorously to make sure they were well incorporated before loading on to the testing plate.

Results and Discussion:

As with all of our consulting and rheological testing, we started with the analysis of a known standard to confirm the calibration and performance of the instrument. Newtonian silicone oil with a nominal viscosity of 12.5 Pa*s at 25°C was tested at room temperature using the same setup. Figure 1 shows the shear viscosity and torque over 22 minutes at a constant shear rate of 1.0 s⁻¹. As expected, all values are within acceptable limits.

Figure 2 shows a typical experiment for the sample labeled "32014" at a constant shear rate of 0.785 s⁻¹. Both shear viscosity and torque decreased gradually in the through out the testing time. Also in Figure 2, the measurement of sample labeled "32014 + type I" was compared with sample "32014". The addition of type-I deicer in the grease sample showed a significant decreased in both viscosity and torque through out the testing time. Two repeated runs of sample labeled "32014+ type I" are shown in Figure 3 and they showed similar trend which confirmed repeatability of the testing methodology.

Figure 4 shows the comparison of the data for sample "83261" and "83261+ type I". Type I deicer showed different effect by increased both viscosity and torque of the sample at the end of testing time. With the addition of type-I deicer, the sample showed a slower decrease in viscosity and torque compare to pure grease sample "83261".

From these results, it clearly demonstrates the capability of our unit to generate the precise data, with a proper method. The time dependence of the shear viscosity provides useful information on the effects of different types of anti-icer mixed with the grease samples.

Steven Colo will follow up with you in the next few days to discuss these results and the next logical step in this project.

Sincerely,

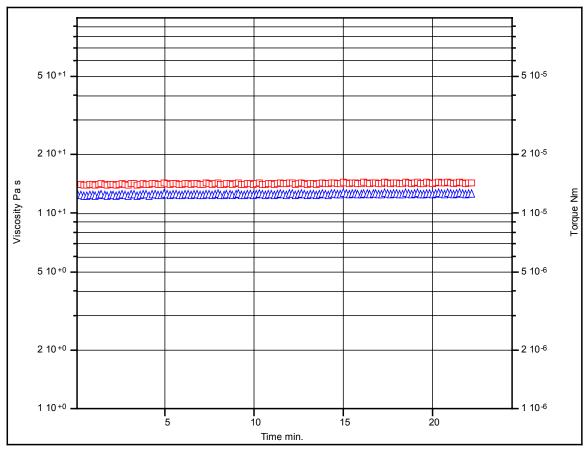
Dr. Tien T. Dao Staff Engineer

E-Mail: info@atsrheosystems.com Visit our Web site @ http://www.atsrheosystems.com





P 15 ETC Gap 1.000 mm Manual control Number of measurements 200 Measurement interval 1.000E+0 s Shear rate table Shear rate 1.000E+0 1/s Delay time 5.000E+0 s Integration time 5.000E+0 s Operator Date 10/28/2010 Sample 13 Pa.



13Pas oil time sweep test.RCO

--- Viscosity

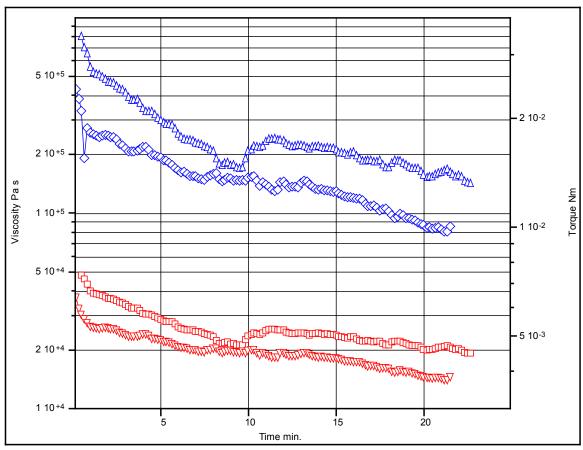
_∆ Torque

Figure 1: Constant shear rate test for a Newtonian oil with nominal viscosity of 12.5 Pa*s at room temperature.





P 15 ETC Gap 1.000 mm Manual control Number of measurements 200 Measurement interval 1.000E+0 s Shear rate table Shear rate 7.850E-1 1/s Delay time 5.000E+0 s Integration time 5.000E+0 s Operator Date 10/28/2010 Sample N



Constant Rate_32014 (Nye Lubricants 374A)_T1_P15.RCO

-U- Viscosity

— Torque

Constant Rate_32014 plus type I t1.RCO

√ Viscosity

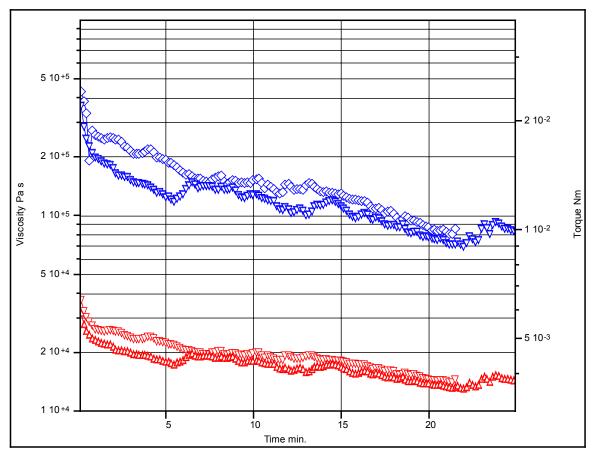
→ Torque

Figure 2: Shear viscosity and torque of the sample labeled "32014 + Type I antiicer" and "32014" at a constant shear rate of 0.785 s⁻¹ under -54 °C.





P 15 ETC Gap 1.000 mm Manual control Number of measurements 200 Measurement interval 1.000E+0 s Shear rate table Shear rate 7.850E-1 1/s Delay time 5.000E+0 s Integration time 5.000E+0 s Operator Date 10/28/2010 Sample



Constant Rate_32014 plus type I t1.RCO

√- Viscosity

→ Torque

Constant Rate_32014 plus type I_T1_P15-latest run.RCO

→ Viscosity

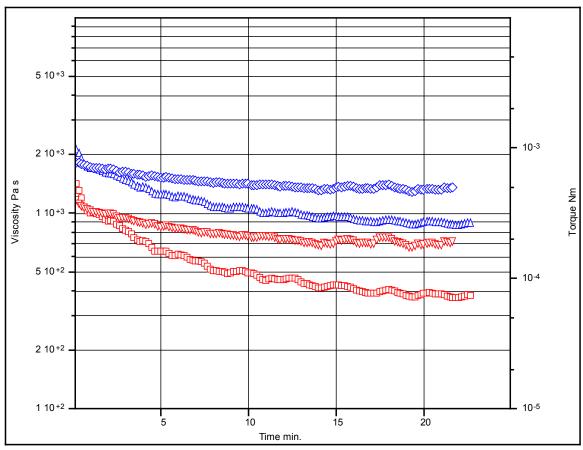
▼- Torque

Figure 3: Repeated tests Shear viscosity and torque of the sample labeled "32015 + type I" at a target shear rate of 0.785 s⁻¹ under -54 °C.





P 15 ETC Gap 1.000 mm Manual control Number of measurements 200 Measurement interval 1.000E+0 s Shear rate table Shear rate 7.850E-1 1/s Delay time 5.000E+0 s Integration time 5.000E+0 s Operator Date 10/28/2010 Sample A



Constant Rate_83261 (Tribolube)_T1_P15.RCO

--- Viscosity

— Torque

Constant Rate_83261 plus type I_T1_P15.RCO

√ Viscosity

→ Torque

Figure 4: Sample labeled "83261 + Type I anti-icer" and sample labeled 83261 at a constant shear rate of 0.785 s⁻¹ under -54 °C.



FINAL REPORT

EVALUATION OF POSSIBLE DEGRADATION IN VISIBILITY AND INCREASED SLIPPERINESS DUE TO RESIDUE FROM USE OF AIRCRAFT DEICING FLUIDS

Comparison of EcoFlo Reduced PG Fluid to Conventional PG Based Fluid for ESTCP Project WP-200905, Demonstrate a Low Biochemical Oxygen Demand Aircraft Deicing Fluid

September 2011

Prepared by: Science Applications International Corporation 4031 Colonel Glenn Highway Beavercreek, OH 45431

Under:

U.S. Army Corps of Engineers, Mobile District Contract No. W91278-09-D-0037 Task Order No. 0007

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Acknowledgements

This project was conducted for the Environmental Security Technology Certification Program (ESTCP) and the US Air Force Aeronautical Systems Center (ASC) by SAIC, The Boeing Company and the Anti-Icing Materials International Laboratory (AMIL) at the University of Quebec at Chicoutimi.

The project manager for the EcoFlo aircraft deicing fluid demonstration project was Ms. Mary Wyderski from the US Air Force Aeronautical Systems Center and the technical lead for the wind tunnel fluid evaluation covered in this report was Mr. Donald Tarazano of SAIC.

This effort was supported by Ms. Arlene Beisswenger of AMIL, Ms. Melissa Tolentino, Mr. Steven Chapel, Mr. Charles Royas, Mr. John Braun and Mr. Alan Lepper of The Boeing Company and Ms. Megan Hawk and Mr. James Davila of SAIC.

Executive Summary

Effectively deicing aircraft to allow operations in adverse weather conditions is critical to the US Air Force. Currently, aircraft deicing fluid (ADF) is a significant environmental problem at airports. This is due to significant depletion of oxygen in receiving waters, caused by ADF runoff and primarily attributable to the propylene glycol (PG) makeup of the fluid.

Previous alternative (reduced biochemical oxygen demand (BOD/COD)) ADF formulations have indicated potential to leave a residue that does not readily flow off the aircraft and can lead to blurred windows and slippery aircraft surfaces (a safety risk for immediate post flight inspections in which personnel may venture on the aircraft wings).

A new alternative ADF formulation, with reduced BOD/COD impact, is now being marketed for commercial aircraft deicing and promises to eliminate any residue issues. The testing described in this report covers a preliminary evaluation of the fluid (EcoFlo by Octagon Process, L.L.C.). This evaluation studied the condition of surfaces exposed to aircraft takeoff speed airflow in a wind tunnel subsequent to the application of the ADF. Transparent surfaces were evaluated for any impact on visual clarity attributable to ADF residue and painted aluminum surfaces were evaluated for slipperiness. In both evaluations, a conventional PG based ADF was used for comparison.

Evaluations indicated no notable impact on visual clarity for either the EcoFlo or the conventional PG based ADF. Both fluids left significantly slippery surfaces after wind tunnel exposure. In some cases, but not all, the EcoFlo appears to be slightly more slippery then the conventional PG ADF, but in all cases, the surfaces were well beyond the threshold of what might be considered a safe walking surface. These results suggest that EcoFlo performs comparably to conventional PG based ADF and its use can be expected to imply no greater risk to aircraft users and maintainers.

1 Introduction

United States Air Force aircraft must be able to fly in adverse weather conditions, and deicing aircraft in adverse weather is a critical component of this requirement. Currently, aircraft deicing fluid (ADF) is a significant environmental problem at airports. The fluids exert a very high Biological Oxygen Demand (BOD) and Chemical Oxygen Demand (COD) on storm water runoff. This leads to oxygen depletion, and can cause adverse effects for aquatic life in receiving waters, and can result in increased processing costs at waste water treatment facilities.

The Department of Defense (DoD), through the Environmental Security Technology Certification Program (ESTCP), is motivated to identify alternatives to high BOD Propylene Glycol (PG) based ADFs and funded a previous ADF demonstration of a fluid developed by Battelle Memorial Institute (ESTCP Project WP-200124). In that demonstration, the aircraft flight crew and maintenance crew observed a persistent residue that was both slippery and impaired visibility though some of the aircraft transparencies.

The fluid has since been reformulated to address the residue issue, and licensed to Octagon Process, L.L.C. (marketing the ADF as EcoFlo). The fluid has been qualified for commercial use as an SAE/AMS 1424 Type I Deicing Fluid, but has not been tested with unique materials and performance requirements associated with military aircraft. A demonstration of performance and environmental benefits of the EcoFlo formulation at Air Force facilities has been funded by ESTCP as Project WP-200905. Prior to the demonstration, and in order to obtain a preliminary assessment of any residue effects with the new fluid formulation, the project team determined that wind tunnel testing might be suitable.

With the cooperation of the Boeing C-17 program and the Anti-icing Materials International Laboratory (AMIL) in Quebec, Canada, a comparative evaluation of EcoFlo and a PG based fluid was carried out in a wind tunnel. Both slipperiness and visual degradation were investigated at various temperatures and concentrations.

2 Methods, Assumptions and Procedures

Previous formulations of PG free ADFs (ESTCP Project WP-200124) have led to observations of degradation in visibility (blurry windows) during flight and excessive slipperiness after the flight (a possible hazard during post flight inspections). These observations were gathered from experienced personnel involved with that demonstration.

Acknowledging that there are no well established test methods for visual degradation or residual slipperiness due to ADF use, the project team determined that testing in a wind tunnel would provide a reasonable indication of fluid performance at aircraft take-off speeds. Furthermore, as there are no specified threshold requirements for visual degradation or slipperiness, it was determined that performance of the EcoFlo would be compared to the performance of a conventional PG based fluid with slipperiness and visual degradation effects that have been found acceptable by the military aircraft community.

To evaluate deicing fluid residue effects, fluids were applied to the floor (or, for slipperiness measurements, to an aluminum panel secured flat on the floor) in the test duct section of a wind

tunnel. The wind tunnel was operated until fluid height measurements indicated 100% fluid elimination and then residue effects on the floor or aluminum panel were investigated.

Degradation in visibility (i.e., the likelihood of the fluid obscuring or blurring windows) was evaluated by observing and photographing an eye chart through the floor of the wind tunnel after a fluid elimination test run.

To evaluate slipperiness, a tribometer designed for measurements on wet surfaces was utilized. The measurements approximate the angle at which a heel strike in a walking individual's step is likely to result in a slip, with more slippery surfaces requiring a shorter stride or a lower impact angle (relative to normal to the surface) to initiate a slip.

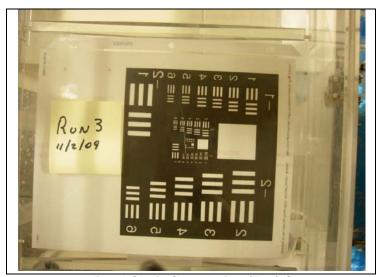
2.1. Equipment/Apparatus

2.1.1. Wind Tunnel

The wind tunnel, operated by AMIL, is compliant with SAE Aerospace Standard AS5900, Standard Test Method for Aerodynamic Acceptance of SAE AMS 1424 and SAE AMS 1428 Aircraft Deicing/Anti-icing Fluids. The wind tunnel contains a temperature controlled test section or "test duct" allowing the observation of fluid elimination from surfaces parallel to the airflow.

2.1.2. Visual Clarity/Resolution (Eye) Chart

To assess the potential for fluid residue to impede visibility for pilots or refueling boom operators by migrating onto and contaminating aircraft transparencies, a simple eye chart allowing a determination of visual clarity or resolution (Figure 1) was affixed to the bottom of the Plexiglas test duct section of the wind tunnel, allowing the impact of any residue remaining within the test duct subsequent to wind tunnel operation to be evaluated.



Visual Clarity/Resolution (Eye) Chart

2.1.3. English XL Variable Incidence Tribometer

The English XL Variable Incidence Tribometer (VIT) (Figure 2) is a portable device featuring a 1.25 inch diameter Neolite (a once common rubber heel and sole material for shoes) test foot. The Neolite disk is mounted on a piston which can be extended with a consistent and repeatable force by regulated feed of pressurized gas from a small CO₂ canister. The operator can vary the impact angle (angle at which the test foot approaches the test surface) using an adjusting knob on the VIT.



English XL Variable Incidence Tribometer (on painted aluminum test panel at AMIL)

Once contact with the surface occurs, the test foot can pivot freely and the piston can rotate about the axis at the top of the device. The measurement of slipperiness provided by the VIT is a Slip Resistance Index (shortened, for convenience, to "Slip Index"). The manufacturer defines the Slip Index as the tangent of the angle between the test foot impact direction and the normal to the test surface. The manufacturer's working range for the Slip Index is 0 to 1, with 0 equivalent to a 0° impact (basically walking in place) and 1 representing a 45° impact (a very large stride).

2.2. Deicing Fluids

For testing, a sample of EcoFlo was provided by Octagon. The fluid is provided as a concentrate, and was diluted with water to at a 65%/35% EcoFlo/water solution.

For comparison, a PG solution, Octagon Octaflo EF, was used, also at a 65/35 dilution ratio with water.

2.3. Assumptions

- Water reduction: During deicing operations, in which heated fluid is sprayed onto aircraft from a distance, some evaporation of water occurs before the fluid reaches the aircraft. In the test scenarios documented in this report, 20% of the water was allowed to evaporate from the test fluids prior to application of the fluids to the wind tunnel test duct in order to simulate the water loss during actual deicing operations. (Note that the fluids were also evaluated without any water reduction)
- Visibility: The ability of airflow to clear residual fluid from aircraft transparencies at various locations on the aircraft and facing various directions relative to the forward motion of the aircraft was estimated by the ability of the airflow to remove fluid from a transparent surface parallel to the airflow in the test duct of the wind tunnel.
- Slip Resistance: The Slip Index per the utilized equipment (English XL VIT) is equal to the tangent of the angle of approach/impact of the test foot preset on the device. It should not be assumed that the angle of impact or the Slip Index is consistent across various test methodologies and available equipment choices.
 - For the English XL VIT, a Slip Index greater than 0.5 (the tangent of approximately 26.6°) is conventionally considered adequate for normal walking.
 - While the tangents (and thus the Slip Index) of impact angles between 26.6° and 0° (Slip Index between 0.5 and 0.0) are fairly linear, the Slip Index scale is not intuitive (i.e., the slipperiness difference between a surface with a Slip Index of 0.2 and one measuring 0.4 is not obvious). For the purposes of this study, it is more suitable to use angle of impact (one can more easily understand the difference between a shortened stride giving an impact angle of 21.8° from vertical, and an even shorter, almost shuffling stride with the heel hitting at 11.3°).

2.4. Evaluation Procedures

2.4.1. Establishing Test Duration

Prior to the initiation of testing, the time required to eliminate deicing fluid from the test duct of the wind tunnel was established. For each anticipated set of test parameters, including fluid concentration (subsequent to water reduction), test temperature, and initial fluid thickness on the test surfaces, the time necessary to reach a fluid thickness of 0mm \pm 0.025 mm while operating the wind tunnel was defined as the time to reach 100% fluid elimination. For the majority of test conditions, that time was 5 to 15 minutes.

2.4.2. Fluid Preparation and Wind Tunnel Operation

The fluid was prepared by mixing with water to the desired concentration, and heating to 60°C, the typical temperature at which fluid is applied in actual deicing operations. For fluid runs reflecting the effects of water reduction, the 65/35 solutions were heated on a hot plate until the

desired amount of the water evaporated (in most cases this was 20% of the water). Final solution concentrations were verified by refractive index measurements.

Wind tunnel operations were essentially the same for both the slipperiness and the visual degradation evaluations. The only exception was that the deicing fluid was applied to a painted aluminum panel placed in the test duct for the slip resistance measurements. Fluid was poured on the test duct floor or to the aluminum panel on the test duct floor, as appropriate, and leveled to the desired fluid thickness. The wind tunnel was operated with an airflow of $65 \text{ m/s} \pm 5 \text{ m/s}$ in the test section (the wind velocity called out in SAE Aerospace Standard 5900 for the High Speed Ramp Test, based on takeoff conditions typical of large transport type jet aircraft) for the time previously established for the given set of test parameters. If, at the end of the test, fluid height measurements did not indicate 100% elimination of the fluid, 5-10 minutes was added to the test duration and the full procedure was repeated for the new, longer duration. Test conditions for both slipperiness and visual degradation are listed in Table 1. For slipperiness, in addition to the conditions below, the painted aluminum panel was tested in a dry state, without exposure to wind tunnel conditions, to establish a baseline for slipperiness of the surface with no fluid contamination.

	_		
Test	(, vu	ditio	ne
1631	CUI	ullu	

Fluid	Water	Wind Tunnel	Initial Fluid
Fluid	Reduction	Temperature	Thickness
			1 mm
EcoFlo	0%	0°C	2 mm
Octaflo EF	20%	-20°C	2 111111
o otano 21	2070	200	4 mm

2.4.3. Visual Degradation Evaluation

To evaluate and document any degradation in clarity due to fluid residue on the test duct floor, after each run the test duct section was opened and a photograph was taken showing the eye chart through the Plexiglas floor. Visual clarity or resolution was evaluated for each set of test conditions in order to compare any degradation effects of the EcoFlo fluid with the PG fluid.

2.4.4. Slipperiness Evaluation

The procedure for use of the English XL VIT involved setting the device at a small angle (i.e., nearly vertical, or normal to the aluminum panel), pressurizing/extending the piston and observing whether or not the test foot slipped. If the foot did not slip, the angle was increased, and the observation repeated. For testing on wet surfaces, the VIT was repositioned, as the test foot would effectively clear the area of contact making subsequent measurements in that specific area unreliable.

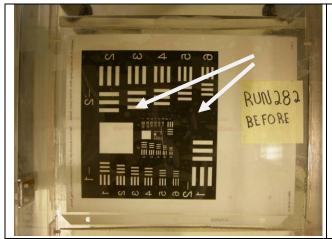
Once the approximate slip angle was established, the operator took additional measurements in an attempt to identify the greatest angle where the foot would not slip and the least angle where the foot would slip. A minimum of eight measurements for each run was planned but in most

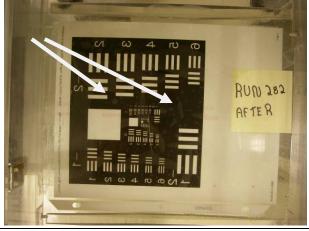
cases eleven or more were performed. (These were slip or no-slip checks at various angles (in an attempt to pinpoint the threshold where slip occurred) rather than repetitions of a measurement of magnitude, and should not be assumed to imply a sample size of statistical significance for each individual run).

3 Results and Discussion

3.1. Visual Degradation

The examination of photos taken before and after operating the wind tunnel to remove fluid from the test duct indicate that for either fluid no significant degradation of resolution or clarity (i.e., no blurriness) can be observed, for any of the test conditions. In some instances, "bubbles" can be seen between the chart and the outside of the Plexiglas wall of the wind tunnel, but those are consistent in the before and after pictures. Figure 3 shows the consistency in both clarity and presence of bubbles (indicated by arrows). Photographs for the various sets of test conditions are organized for comparison in Appendix A.



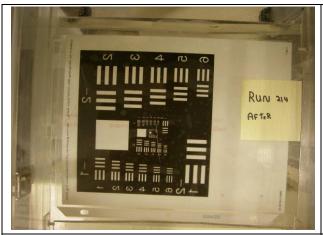


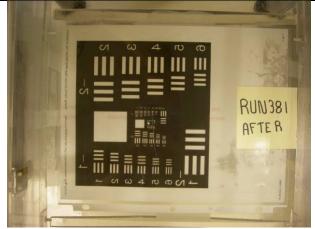
Wind Tunnel Temperature: 0°C H₂O Reduction Before Test; 20% Initial Fluid Thickness: 1 mm

Wind Tunnel Temperature: 0°C H₂O Reduction Before Test; 20% Initial Fluid Thickness: 1 mm

Before and After Photos Showing Air Bubbles (EcoFlo Fluid Test)

While the wind tunnel configuration may not duplicate all conditions for transparencies on military aircraft (e.g., the parallel airflow in the wind tunnel test section is likely not similar to the airflow experienced by a boom operator's window on a KC-135), it should be noted that when a uniform residual thickness of fluid was detected and the test rerun for a longer duration, there is no significant difference in clarity of the eye chart in the photos. This can be seen in the photograph for Run 214, in which residual fluid was detected in the test duct after a 5 minute test run, in comparison to the photograph for Run 381, in which there was no measurable fluid thickness after a 10 minute run (Figure 4). This indicates that a measurable but uniform thickness of fluid seems to have no impact on visual resolution.





Foot

Wind Tunnel Temperature: 0°C H₂O Reduction Before Test; 0% Initial Fluid Thickness: 1 mm

Wind Tunnel Run Time: 5 minutes (Fluid Detected)

Wind Tunnel Temperature: 0°C H₂O Reduction Before Test; 0% Initial Fluid Thickness: 1 mm Wind Tunnel Run Time: 10 minutes

Visual Comparison Between Some Fluid / No Fluid Remaining (EcoFlo Fluid Test)

3.2. Slipperiness

As stated in the equipment description, the VIT provides a Slip Index equivalent to the tangent of the angle between the test foot impact direction and the normal to the test surface. Aircraft deicing fluids tend to make surfaces slippery, and a surface contaminated with fluid residue can be expected to generate a slip at a VIT impact angle less than 26.6° (a Slip Index less than 0.5, i.e., an unsafe walking surface).

For an even more intuitive albeit hypothetical approach, angle of impact can be converted to the stride length. This calculation, however, ignores variations in the mechanics of individual gaits, but can still present another indication of slipperiness differences assuming other variables are held constant. For example if the gait is simplified so that the individual's hip is centered between the two feet when the front foot first impacts, the stride length can be easily calculated (Figure 5).

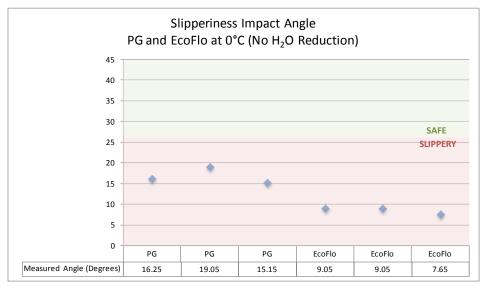
Hip Angle of Impact Stride Length $(L_s) \approx 2 x (h x sin\Theta)$ Where: h = hip height Θ = angle of impact Stride Length

Stride Length Approximation

In this situation, assuming a hip height (h) of 34 inches, heel impact angles (Θ) of 26.6°, 21.8° and 11.3° (Slip Indices of 0.5, 0.4 and 0.2) would imply that the surfaces are unsafe when the stride length (L_S) nears approximately 30 in, 25 in and 13 in, respectively.

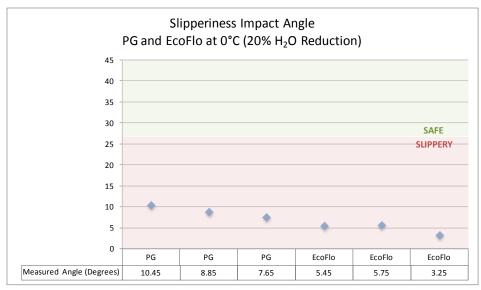
This stride length calculation depends upon an oversimplification of actual walking. In this report, the VIT angle of impact will be used for analysis while calculated stride length will only be used for illustrative purposes.

Time limitations prevented gathering a significant number of repetitions for each set of conditions but the measurements are still fairly consistent. At 0°C, with the fluid at either the selected operational, in-truck concentration (65%vol fluid in water) or at the reduced water concentration, the impact angle at which a slip occurred for EcoFlo was measurably lower than the angle for PG (i.e., the EcoFlo test panel was more slippery than the PG fluid test panel) (Figures 6 and 7).



Slip Measurements at 0°C, with No H₂O Reduction Prior to Test

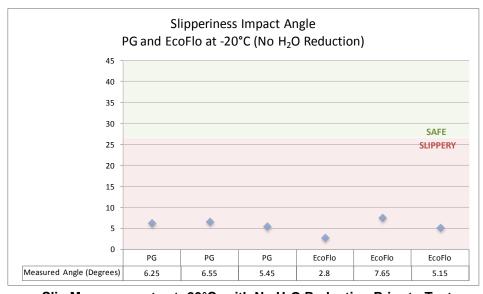
With no water reduction from the fluid before application and wind tunnel operation, the average of the three measurements resulting from the PG was 16.8° (or an estimated stride length of 19.7 in.) while EcoFlo resulted in an average of 8.6° (estimated stride length of 10.2 in.). The EcoFlo treated surface would require a smaller stride for prevention of sliding than the PG, but both fluids are definitely unsafe for normal walking.



Slip Measurements at 0°C, with 20% H₂O Reduction Prior to Test

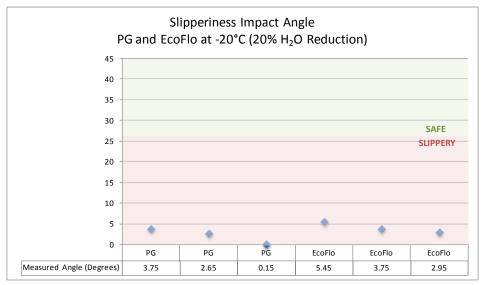
When the fluids were more concentrated by allowing 20% of the water to evaporate prior to application, the slipperiness increased. For PG, the resulting average angle where slip first occurred was 9.0° (estimated stride length of 10.6 in.) while for EcoFlo the average angle was 4.8° (estimated stride length of 5.7 in.). So any residue resulting from EcoFlo was still slightly more slippery than that from PG. (Note that, as an approximate baseline, a test panel with no fluid used for VIT calibration runs at 0°C resulted in slip at just over 31° or an estimated stride length of over 35 in).

After operating the wind tunnel at -20°C, both fluids left the aluminum test panel much more slippery than the 0°C test runs and well beyond what would be considered safe (Figures 8 and 9).



Slip Measurements at -20°C, with No H₂O Reduction Prior to Test

With no water reduction, the average of the three PG measurements was 5.5° (or an estimated stride length of 6.5 in.) while EcoFlo resulted in an average of 5.2° (estimated stride length of 6.1 in.). In this case, the difference between the two fluids is likely negligible compared to the tolerance limitations of the measurement methodology and given the limited number of repetitions. Both fluids result in a highly slippery and clearly unsafe walking surface.



Slip Measurements at -20°C, with 20% H₂O Reduction Prior to Test

When concentrated by removing 20% of the water content, the slipperiness again increased. In this case, the PG fluid resulted in the lower slip angle, 0.2° (estimated stride length of 0.2 in.) in comparison to 3.0° (estimated stride length 3.5 in.) for EcoFlo. Again, the difference between the two fluids is likely negligible compared to the tolerance limitations of the evaluation, but it's clear that both fluids result in a highly slippery surface. (In this case, at -20° C, test panels with no fluid used for VIT calibration runs resulted in slip at slightly smaller angles than the 0° C runs: $25-29^{\circ}$ or an estimated stride length of about 30 in).

Raw data for all English VIT slip evaluation measurements is included in Appendix B.

4 Conclusions & Recommendations

In both evaluations, EcoFlo and Octaflo EF exhibited somewhat similar performance.

For the test conditions and available configurations, visibility through the wind tunnel wall was not discernibly impacted by either fluid. A simulation of complex configurations or aerodynamically quiet areas was, however, beyond the scope of this testing. The results for visual testing, suggesting a lack of extreme contamination issues impacting visibility through transparencies, indicate that the fluid is likely to perform acceptably in field evaluations and eventual operational implementation.

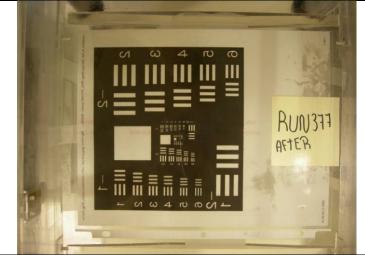
In slipperiness testing, EcoFlo was indicated to be slightly more slippery than the PG fluid when tested at 0°C, and the Octaflo EF was just as, or even more slippery at -20°C. It's not clear that the measured differences between the fluids would be easily discernible by operational personnel attempting to walk on surfaces similar to the test panels (such as aircraft wings), given that any potential residue from either fluid would likely leave the surface extremely slippery. Also, considering that the slipperiness of PG based fluids approaches the slipperiness of EcoFlo as the temperature drops, if personnel have already developed some level of comfort working with Octaflo EF (or a similar PG based ADF) at lower temperatures (where the surfaces left after either fluid test were most slippery), they should be able to adapt to working with the EcoFlo.

The ultimate objective of this testing was to produce a degree of comfort that the EcoFlo ADF would not significantly impact visibility through aircraft transparencies during flight and slipperiness on aircraft surfaces subsequent to flight, especially in comparison to currently used PG based fluids. Given the performance of EcoFlo further evaluation through field testing is merited.

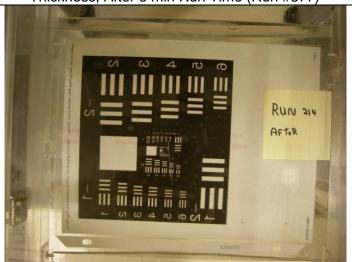
APPENDIX A – VISUAL DEGRADATION EVALUATION PHOTOGRAPHS

			l	I D 627D		l		
				LB-627D SECTION: 2.0 Part E				
				SECTION. 2.0 Fait L			Test	
Test Fluid:	EcoFlo		NON	- GLYCOL BASED F	LUID		Type:	Visual
Tunnel Floor:	Plexiglas			AS RUN SCHEDULE				
PHASE	RUN NUMBER ¹	AMIL Run Number	TUNNEL TEMPERATURE ²	TARGET FLUID TEMPERATURE	% WATER REDUCTION	FLUID THICKNESS	RUN TIME	COMMENTS
SECTION: 2.0		DD\(4.007	0.11.00.0	N/A	.	News	D 4	0.17
PART: B	54	DRYA697	Cold: 0° C	N/A	Dry	None	Basic ⁴	Cal (no photo)
Cold: 0° C 2	214	1059H01A1	Cold: 0° C	Hot: 60°C (140°F) 7	Nominal	1 mm	5 min	Visual
EcoFlo ⁶	381	1059H01A2				1 mm	10 min	Visual/ Focus
	217	1059H02A1				2 mm ⁵	5 min	Visual
	220	1059H04A1				4 mm	5 min	Visual
	382 223	I059H04A2 DRYA728	Cold: 0° C	N/A	Desc	4 mm None	10 min Basic ⁴	Visual/ Focus Cal (no photo)
	223	DRTA/20	Cold: U C	N/A	Dry	None	Dasic	Car (no prioto)
	281	DRYA741	Cold: 0° C	N/A	Dry	None	Basic ⁴	Cal (no photo)
	282	1059H21A1	Cold: 0° C	Hot: 60°C (140°F) 7	20%	1 mm	5 min	Visual
	285	1059H22A1				2 mm ⁵	5 min	Visual
	288	1059H24A1				4 mm	5 min	Visual
	291	DRYA742	Cold: 0° C	N/A	Dry	None	Basic ⁴	Cal (no photo)
	292	l059H41A1	Cold: 0° C	Hot: 60°C (140°F) 7	40%	1 mm	5 min	Visual
	295	1059H42A1				2 mm ⁵	5 min	Visual
	298	1059H44A1	0.11.00.0	N/A	.	4 mm	5 min	Visual
	301	DRYA743	Cold: 0° C	N/A	Dry	None	Basic ⁴	Cal (no photo)
							1	
Cold: -20° C ² EcoFlo ⁶	259	DRYE734	Cold: -20° C	N/A	Dry	None	Basic ⁴	Cal (no photo)
ECOFIO	260	I050H01E1	Cold: -20° C	Hot: 60°C (140°F) ⁷	Nominal	1 mm	5 min	Visual
	389	1059H01E2	Cold20 C	1101. 00 0 (1401)	Nominal	1 mm	15 min	Visual/ Focus
	263	1059H02E1				2 mm ⁵	15 min	Visual
	266	1059H04E1				4 mm	15 min	Visual
	390	1059H04E2			_	4 mm	15 min	Visual/ Focus
	269	DRYE735	Cold: -20° C	N/A	Dry	None	Basic⁴	Cal (no photo)
	323	DRYE747	Cold: -20° C	N/A	Dry	None	Basic ⁴	Cal (no photo)
	324	1059H21E1	Cold: -20° C	Hot: 60°C (140°F) 7	20%	1 mm	15 min	Visual
	327	1059H22E1				2 mm ⁵	15 min	Visual
	330	1059H24E1				4 mm	15 min	Visual
	333	DRYE748	Cold: -20° C	N/A	Dry	None	Basic ⁴	Cal Visual
	334	I059H41E1	Cold: -20° C	Hot: 60°C (140°F) ⁷	40%	1 mm	15 min	Visual
	335	1059H41E2				1 mm	25 min	
	336	1059H41E3				1 mm	1 min	Repeat (no photo)
	337 340	1059H42E1 1059H44E1				2 mm ⁵ 4 mm	25 min 25 min	Visual Visual
	340	IOUSI IHHE I				7 111111	اااااا	viouai
		 		-				
Notes:	—							
				ch run during the test.		2.2		
				per SAE AS5900 Required per SAE AS5				
J.	raiget cold l	iuiu terriperai	ure (± 5 C) as it	Squired per SME ASS	ooo nev A. pala	giapiro.i		
4.	As required	per SAE AS	5900 Rev A. para	graph 5.1				
-		•		000 Rev A. paragraph	15.2.5			
6.	Type I non/lo	w-glycol base	ed fluid.					
7.	Target hot flu	uid temperatu	re (± 5° C)					

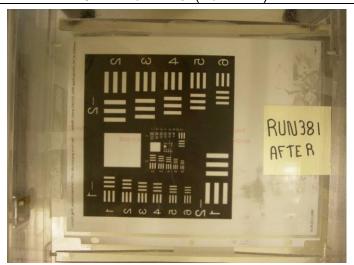
				LB-627D				
				SECTION: 3.0				
				0201101111010				
							Test	
Test Fluid:	Propylene G	ilycol (PG)	NON	- GLYCOL BASED F	LUID		Type:	Visual
Tunnel Floor:	• •	, , ,		AS RUN SCHEDULE				
	J							
PHASE	RUN NUMBER ¹	AMIL Run Number	TUNNEL TEMPERATURE ²	TARGET FLUID TEMPERATURE	% WATER REDUCTION	FLUID THICKNESS	RUN Time	COMMENT
ECTION: 3.0							-	
ART: B								
ANI. D								
old: 0° C ²	376	DRYA756	Cold: 0° C	N/A	Dry	None	Nom	Cal (no photo)
	0.0	51171100	00 0	1471	2.,			car (no prioto)
	377	I061H01A1	Cold: 0° C	Hot: 60°C (140°F) 7	Nominal	1 mm	5 min	Visual
	378	I061H04A1				4 mm	5 min	Visual
	379	1061H21A1	Cold: 0° C	Hot: 60°C (140°F) ⁷	20%	1 mm	5 min	Visual
	380	1061H24A1	00.0.0	1101. 00 0 (140 1)	2070	4 mm	5 min	Visual
	000	10011121711				7	0 111111	Vioudi
	383	DRYA757	Cold: 0° C	N/A	Dry	None	Nom	Cal (no photo)
							+	
old: -20° C ²	384	DRYE758	Cold: -20° C	N/A	Dry	None	Nom	Cal (no photo)
	385	I061H01F1	Cold: -20° C	Hot: 60°C (140°F) ⁷	Nominal	1 mm	10 min	Visual
	344	1061H04E1	00Iu20 0	1101. 00 0 (140 1)	Nominal	4 mm	10 min	Visual
	386	1061H04E2		1		4 mm	10 min	Visual
		1001110122				7 11111	10 111111	Vioudi
	387	I061H21E1	Cold: -20° C	Hot: 60°C (140°F) 7	20%	1 mm	10 min	Visual
	388	l061H24E1		,		4 mm	10 min	Visual
	391	DRY	Cold: -20° C	N/A	Dry	None	Nom	Cal (no photo)
Notes:								
1.	This is the fi	nal run numbe	er assigned to ea	ch run during the test.				
				per SAE AS5900 R				
				equired per SAE AS5		graph 5.1. Not ເ	use in this	s section.
				igraph 5.1. Not use i				
			•	000 Rev A. paragraph	15.2.5			
		lene glycol ba						
7.	Target hot flu	uid temperatu	re (± 5° C)					



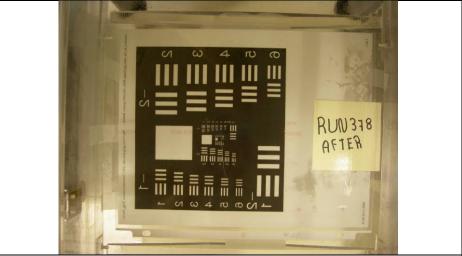
OCTAFLO EF @ 0°C, 0%H₂O Reduction, 1mm Initial Fluid Thickness, After 5 min Run Time (Run #377)



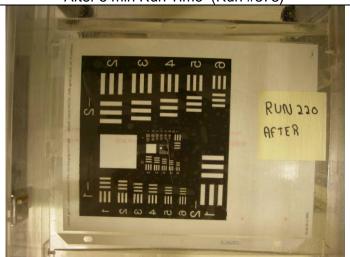
EcoFlo @ 0°C, 0%H₂O Reduction, 1mm Initial Fluid Thickness, After 5 min Run Time (Run #214)



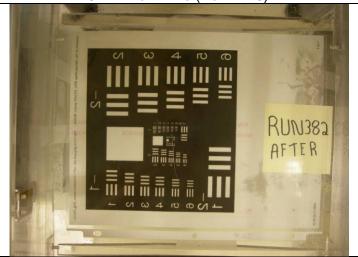
EcoFlo @ 0°C, 0%H₂O Reduction, 1mm Initial Fluid Thickness, After 10 min Run Time (Run #381)



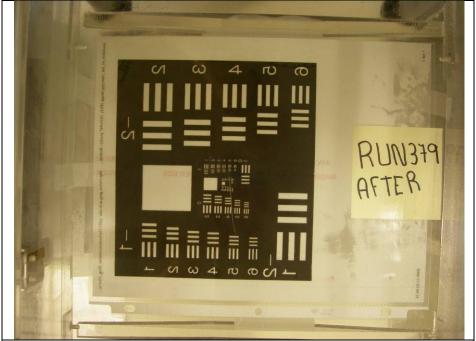
OCTAFLO EF @ 0°C, 0%H₂O Reduction, 4mm Initial Fluid Thickness, After 5 min Run Time (Run #378)



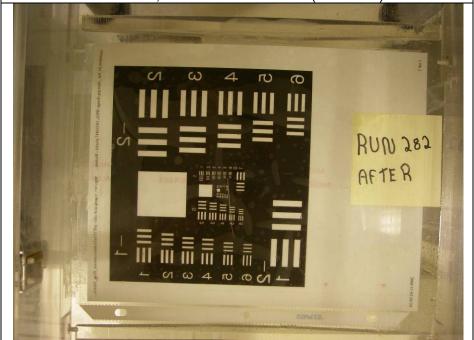
EcoFlo @ 0°C, 0%H₂O Reduction, 4mm Initial Fluid Thickness, After 5 min Run Time (Run #220)



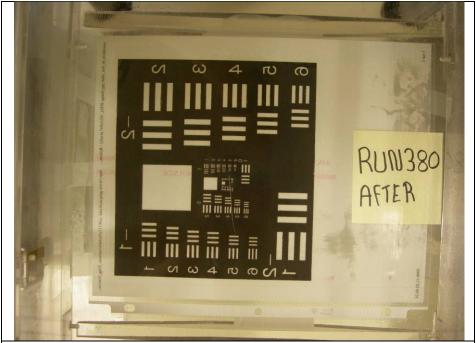
EcoFlo @ 0°C, 0%H₂O Reduction, 4mm Initial Fluid Thickness, After 5 min Run Time (Run #382)



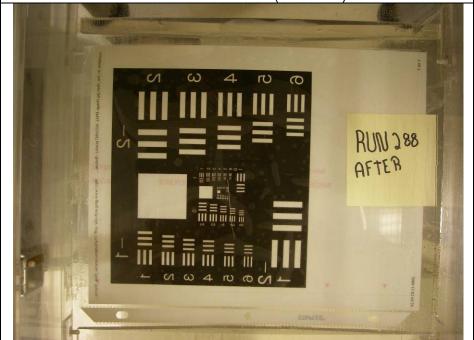
OCTAFLO EF @ 0°C, 20%H₂O Reduction, 1mm Initial Fluid Thickness, After 5 min Run Time (Run #379)



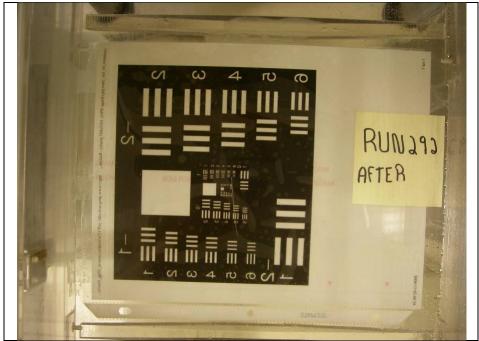
EcoFlo @ 0°C, 20%H₂O Reduction, 1mm Initial Fluid Thickness, After 5 min Run Time (Run #282)



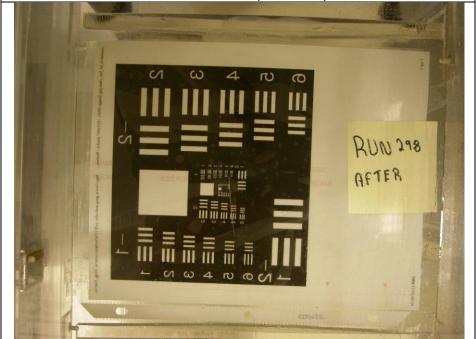
OCTAFLO EF @ 0°C, 0%H₂O Reduction, 4mm Initial Fluid Thickness, After 5 min Run Time (Run #380)



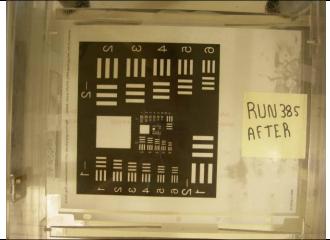
EcoFlo @ 20°C, 0%H₂O Reduction, 4mm Initial Fluid Thickness, After 5 min Run Time (Run #288)



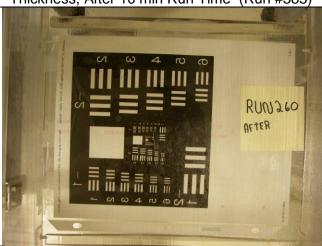
EcoFlo @ 0°C, 40%H₂O Reduction, 1mm Initial Fluid Thickness, After 5 min Run Time (Run #292)



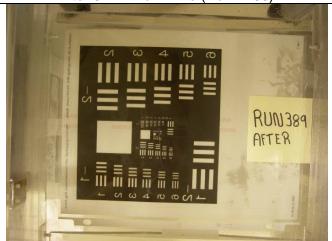
EcoFlo @ 0°C, 40%H₂O Reduction, 4mm Initial Fluid Thickness, After 5 min Run Time (Run #298)



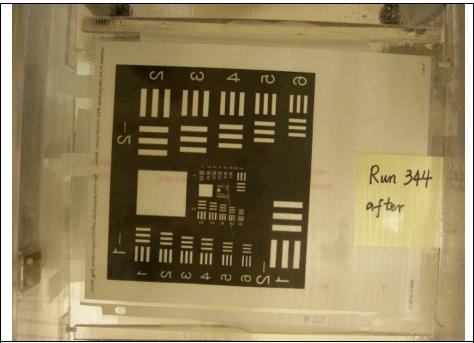
OCTAFLO EF @ -20°C, Nominal%H₂O Reduction, 1mm Initial Fluid Thickness, After 10 min Run Time (Run #385)



EcoFlo @ -20°C, NominalH₂O Reduction, 1mm Initial Fluid Thickness, After 5 min Run Time (Run #260)



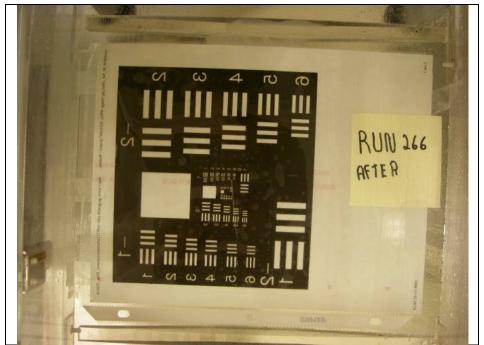
EcoFlo @ -20°C, NominalH2O Reduction, 1mm Initial Fluid Thickness, After 15 min Run Time (Run #389)



OCTAFLO EF @ -20°C, Nominal%H₂O Reduction, 4mm Initial Fluid Thickness, After 10 min Run Time (Run #344)



OCTAFLO EF @ -20°C, Nominal%H₂O Reduction, 4mm Initial Fluid Thickness, After 10 min Run Time (Run #386)



EcoFlo @ -20°C, NominalH2O Reduction, 4mm Initial Fluid Thickness, After 15 min Run Time (Run #266)



EcoFlo @ -20°C, NominalH2O Reduction, 4mm Initial Fluid Thickness, After 15 min Run Time (Run #390)

APPENDIX B – WIND TUNNEL TEST CONDITIONS AND SLIP MEASUREMENT DATA

				LB-627D				
				SECTION: 4.0				
							Test	
	Propylene G	Blycol (PG)		- GLYCOL BASED F			Туре:	Slipperiness
Tunnel Floor:	Aluminum			AS RUN SCHEDULE				
	51111							
PHASE	RUN NUMBER ¹	AMIL Run Number	TUNNEL TEMPERATURE ²	TARGET FLUID TEMPERATURE	% WATER REDUCTION	FLUID THICKNESS	RUN TIME	COMMENTS
Tunnel			Cold: 0° C	None	Dry			
Check Out								
ECTION: 4.0							+	
ART: B	345	DRYA750	Cold: 0° C	N/A	Dry	None	Calibration	Slipperiness
old: 0° C ²								
	346	l061H01A1	Cold: 0° C	Hot: 60°C (140°F) 7	Nominal	1 mm	5 min	Slipperiness
	347	l061H02A1				2 mm ⁵	5 min	Slipperiness
	348	I061H04A1				4 mm	5 min	Slipperiness
	349	I061H21A1	Cold: 0° C	Hot: 60°C (140°F) 7	20%	1 mm	5 min	Slipperiness
	350	I061H22A1				2 mm ⁵	5 min	Slipperiness
	351	l061H24A1				4 mm	5 min	Slipperiness
old: -20° C ²	200	DRYE753	C-14: 20° C	N/A	D	Nama	O-libti	Olimanada
old: -20° C -	360	DRYE/53	Cold: -20° C	N/A	Dry	None	Calibration	Slipperiness
	361	I061H01E1	Cold: -20° C	Hot: 60°C (140°F) 7	Nominal	1 mm	10 min	Slipperiness
	364	I061H01E2		` ,		1 mm	10 min	Slipperiness
	362	l061H02E1				2 mm ⁵	10 min	Slipperiness
	363	I061H04E1				4 mm	10 min	Slipperiness
	205	1004110454		Hat 0000 (4400E) 7			10 1	Olimanada
	365		Cold: -20° C	Hot: 60°C (140°F) ⁷	20%	1 mm	10 min	Slipperiness
	366 367	I061H22E1 I061H24E1				2 mm ⁵	10 min	Slipperiness
	367	1001112461				4 mm	10 min	Slipperiness
		+					+	
							1	
Notes:								
				ch run during the test				
				per SAE AS5900 R				
3.	Target cold	fluid temperat	ture (± 3° C) as re	equired per SAE AS5	900 Rev A. para	graph 5.1. Not ເ	use in this	section.
4.	As required	per SAE AS	5900 Rev A. para	graph 5.1. Not use i	n this section.			
				00 Rev A. paragraph	15.2.5			
6.	Type I propy	lene glycol ba	ased fluid.					
		uid temperatu						

				LB-627D				
				SECTION: 5.0				
				020.1014.0.0			Test	
Test Fluid:				- GLYCOL BASED F			Type:	Slipperiness
Tunnel Floor:	Aluminum			AS RUN SCHEDULE				
PHASE	RUN NUMBER ¹	AMIL Run Number	TUNNEL TEMPERATURE ²	TARGET FLUID TEMPERATURE	% WATER REDUCTION	FLUID THICKNESS	RUN TIME	COMMENT
FIIAGE	HOMBER	Number	TENT EXATORE	TEMI EXATORE	KEDOOTION	THIOTHELOO	111111	COMMENT
Tunnel Check Out			Cold: 0° C	None	Dry			
ECTION: 5.0								
ART: B	352	DRYA751	Cold: 0° C	N/A	Dry	None	Calibration	Slipperiness
old: 0° C 2								
	353	1059H01A1	Cold: 0° C	Hot: 60°C (140°F) ⁷	Nominal	1 mm	5 min	Slipperiness
	354	1059H02A1				2 mm ⁵	5 min	Slipperiness
	355	I059H04A1				4 mm	5 min	Slipperiness
	356	1059H21A1	Cold: 0° C	Hot: 60°C (140°F) 7	20%	1 mm	5 min	Slipperiness
	357	I059H22A1	-	, ,		2 mm ⁵	5 min	Slipperiness
	358	1059H24A1				4 mm	5 min	Slipperiness
	359	DRYA752					Basic	No slip data
old: -20° C ²	368	DRYE754	Cold:-20° C	N/A	Dry	None	Calibration	Slipperiness
	369	I059H01F1	Cold: -20° C	Hot: 60°C (140°F) ⁷	Nominal	1 mm	15 min	Slipperiness
	370	1059H02E1	00ia. 20 0	(11011)	Nomman	2 mm ⁵	15 min	Slipperiness
	371	I059H04E1				4 mm	15 min	Slipperiness
	372		Cold: -20° C	Hot: 60°C (140°F) 7	20%	1 mm	15 min	Slipperiness
	373	I059H22E1				2 mm ⁵	15 min	Slipperiness
	374	I059H24E1				4 mm	15 min	Slipperiness
	375	DRYE755					Basic	No slip data
Notes:								
				ch run during the test				
				per SAE AS5900 R				
				equired per SAE AS5		graph 5.1. Not ι	ise in this	section.
				ıgraph 5.1. Not use i				
				000 Rev A. paragraph	5.2.5			
		ow-glycol base						
7.	Target hot fl	uid temperatu	re (± 5° C)					

Run Number:	345						
Comments:	Dry, no fluid.						
	Slips between 32.5 and 35 deg						
	Average = 33.8 deg / Slip Index = 0.67						
	_	•					
			.				
Measurement	Slip Data	Slip Index	Did device slip?				
Number	(deg.)		(yes/no)				
1	30.0	0.58	no				
2		0.64	no				
3		0.70	yes				
4		0.70	no				
5		0.70	yes				
6		0.77	yes				
7	25.0	0.47	no				
8		0.52	no				
9		0.58	no				
10	30.3	0.58	no				
11	30.6	0.59	no				
12	30.9	0.60	no				
13	35.0	0.70	yes				
14	35.3	0.71	yes				

Run Number:	347					
Comments:	PG, residue visible, fluid streaking noted.					
	Slips between 18.1 and 20 deg					
	Average = 19.1 deg / Slip Index = 0.35					
Measurement	Slip Data	Slip Index	Did device slip?			
Number	(deg.)	Onp macx	(yes/no)			
Number	(ucg.)		(yearle)			
1	15.0	0.27	no			
2	20.0	0.36	no			
3	22.5	0.41	no			
4	25.0	0.47	no			
5	27.5	0.52	no			
6	30.0	0.58	yes			
7	20.0	0.36	no			
8	22.5	0.41	no			
9	25.0	0.47	no			
10	27.5	0.52	yes			
11	22.5	0.41	yes			
12	17.5	0.32	no			
13	17.8	0.32	no			
14	18.1	0.33	no			
15	20.0	0.36	yes			
16	20.3	0.37	yes			
17	20.6	0.38	yes			

Run Number:	346					
Comments:	PG, residue visible, fluid streaking noted.					
	Slips between 17.5 and 20 deg Average = 18.8 deg / Slip Index = 0.34					
Measurement	Slip Data	Slip Index	Did device slip			
Number	(deg.)	onp muex	(yes/no)			
Nullibei	(ueg.)		(yes/110)			
1	10.0	0.18	no			
2	12.5	0.22	nc			
3	15.0	0.27	no			
4	17.5	0.32	no			
5	20.0	0.36	no			
6	25.0	0.47	no			
7	27.5	0.52	no			
8	30.0	0.58	yes			
9	32.5	0.64	yes			
10	35.0	0.70	yes			
11	20.0	0.36	,			
12		0.32	yes			
13		0.18	no			
14		0.27	no			
15		0.32				
16		0.36	no			
17		0.41	yes			
18		0.36	,			
19	-	0.22	no			
20		0.27	no			
21	17.5	0.32	yes			

Run Number:	348					
Comments:	PG, residue visible, fluid streaking noted. Slips between 10.9 and 15.3 deg Average = 13.1 deg / Slip Index = 0.23					
Measurement	Slip Data	Slip Index	Did device slip			
Number	(deg.)	onp muex	(yes/no)			
Number	(ueg.)		(yes/110)			
1	20.0	0.36	nc			
2	25.0	0.47	no			
3	27.5	0.52	ves			
4	30.0	0.58	yes			
5	17.5	0.32	yes			
6	12.5	0.22	yes			
7	7.5	0.13	nc			
8	10.0	0.18	nc			
9	12.5	0.22	yes			
10	7.5	0.13	nc			
11	10.0	0.18	nc			
12	10.3	0.18	nc			
13	10.6	0.19	nc			
14	10.9	0.19	nc			
15	15.0	0.27	nc			
16	15.3	0.27	yes			
17	15.6	0.28	yes			
18	20.0	0.36	yes			

Run Number:	349					
Comments:	PG, residue visible (slightly more than in prior ru					
	fluid streaking noted.					
	Slips between 1					
	Average = 10.5	deg / Slip Index	= 0.18			
Measurement	Slip Data	Slip Index	Did device slip?			
Number	(deg.)	Slip Illuex	(yes/no)			
Number	(deg.)		(yes/110)			
1	20.0	0.36	no			
2	22.5	0.41	no			
3	25.0	0.47	yes			
4	27.5	0.52	yes			
5	15.0	0.27	yes			
6	7.5	0.13	no			
7	10.0	0.18	no			
8	10.3	0.18	no			
9	10.6	0.19	yes			
10	10.9	0.19	yes			
11	15.0	0.27	yes			

Run Number:	351					
Comments:	PG, residue visible (similar to Run 349),					
	fluid streaking n					
	Slips between 7					
	Average = 8.0 de	eg / Slip Index =	0.14			
Measurement	Slip Data	Slip Index	Did device slip?			
Number	(deg.)		(yes/no)			
	(***3)		0 ,			
1	15.0	0.27	no			
2	17.5	0.32	yes			
3	20.0	0.36	yes			
4	10.0	0.18	yes			
5	5.0	0.09	no			
6	7.5	0.13	no			
7	7.8	0.14	no			
8	8.1	0.14	yes			
9	8.4	0.15	yes			
10	12.5	0.22	yes			
11	5.0	0.09	no			
12	7.5	0.13	no			
13	7.8	0.14	yes			
14	10.0	0.18	yes			

Run Number:	350		
Comments:	PG, residue visi	ın 349),	
	fluid streaking n	oted.	
	Slips between 8	.7 and 9.0 deg	
	Average = 8.9 de	eg / Slip Index =	0.16
Measurement	Slip Data	Slip Index	Did device slip?
Number	(deg.)		(yes/no)
1	45.0	0.27	
2	15.0 17.5	0.27	no
3		0.32	no
3	20.0 22.5	0.36	yes
5	10.0	0.41	yes no
6	10.3	0.18	yes
7	7.5	0.13	no
8	7.8	0.13	no
9	8.1	0.14	no
10	8.4	0.15	no
11	8.7	0.15	no
12	9.0	0.16	yes
13	15.0	0.27	yes
			,

Run Number: Comments:	352 Dry, no fluid.					
	Slips between 30.9 and 31.2 deg Average = 31.1 deg / Slip Index = 0.60					
Measurement	Slip Data	Slip Index	Did device slip?			
Number	(deg.)		(yes/no)			
1	15.0	0.27	no			
2	20.0	0.36	no			
3	25.0	0.47	no			
4	30.0	0.58	no			
5	30.3	0.58	no			
6	30.6	0.59	no			
•	30.9	0.60	no			
8	31.2	0.61	yes			
9	37.5	0.77	yes			
10	25.0	0.47	no			
12	27.5 30.0	0.52	no			
13	30.0	0.58 0.64	no			
13	32.5	0.64	yes			

Run Number:	353			
Comments:	EcoFlo Residue appears shiny & slick, no streaks			
	CO2 cartridge ran out after last point.			
	Slips between 8	Slips between 8.1 and 10 deg		
	Average = 9.1 de	eg / Slip Index =	0.16	
Measurement	Clin Data	Clim Impless	Did device slip?	
	Slip Data	Slip Index	(yes/no)	
Number	(deg.)		(yes/no)	
1	5.0	0.09	no	
2	7.5	0.13	no	
3	10.0	0.18	no	
4	12.5	0.22	no	
5	15.0	0.27	no,slide	
6	15.3	0.27	yes	
7	15.6	0.28	yes	
8	17.5	0.32	yes	
9	7.5	0.13	no	
10	10.0	0.18	yes	
11	5.0	0.09	no	
12	7.5	0.13	no	
13	7.8	0.14	no	
14	8.1	0.14	no	

Run Number: Comments:	355 EcoFlo Residue appears shiny & slick, no strea Slips between 7.5 and 7.8 deg Average = 7.7 deg / Slip Index = 0.16 t Slip Data Slip Index Did device sli			
Number	(deg.)		(yes/no)	
1	5.0	0.09	no	
2	7.5	0.13		
3	10.0	0.18	yes	
4	12.5	0.22	yes	
5	5.0	0.09		
7	7.5	0.13 0.14	no	
8	7.8 8.1	0.14	yes	
9	5.0	0.14	yes	
10	7.5	0.09	no no	
	7.0	0.10	2	

Run Number:	354				
Comments:	EcoFlo Residue appears shiny & slick, no streaks				
	Slips between 8.1 and 10 deg				
	Average = 9.1 de	eg / Slip Index =	0.16		
Measurement	Slip Data	Slip Index	Did device slip?		
Number	(deg.)		(yes/no)		
	40.0	0.10			
1	10.0	0.18	no		
2	12.5	0.22	no		
3	15.0	0.27	no		
5	17.5	0.32	yes		
6	20.0 7.5	0.36 0.13	,		
7	7.5 10.0		no		
8	10.0	0.18 0.22	no ves		
9	7.5	0.22	yes no		
10	10.0	0.13	ves		
11	10.0	0.18			
12	7.5	0.18	yes no		
13	7.8	0.13	no		
14	8.1	0.14	no		
15	10.0	0.14	ves		
"	10.0	0.10	yes		

Run Number: Comments:	EcoFlo Residue appears shiny & slick, no str Residue appears thicker than in previous run Slips between 5.3 and 5.6 deg Average = 5.5 deg / Slip Index = 0.10			
Measurement	Slip Data	Slip Index	Did device slip?	
Number	(deg.)	-	(yes/no)	
1	5.0	0.09	no	
2	7.5	0.13	no	
3	10.0	0.18	yes	
4	12.5	0.22	yes	
5	7.5	0.13	,	
6	5.0	0.09	no	
7	7.5	0.13	,	
8	5.0	0.09	no	
9	5.3	0.09	no	
10	5.6	0.10	,	
11	5.9	0.10	,	
12	5.0 5.3	0.09 0.09	no	
13	5.5	0.09	no	

Run Number:	357			
Comments:	EcoFlo Residue appears shiny & slick, no streaks			
	evenly distributed, similar to previous run.			
	Slips between 5.6 and 5.9 deg			
	Average = 5.8 de	eg / Slip Index =	0.10	
		<u></u>		
Measurement	Slip Data	Slip Index	Did device slip?	
Number	(deg.)		(yes/no)	
1	5.0	0.09	no	
2	7.5	0.09	no	
3	7.8	0.13	no	
4	8.1	0.14	no	
5	12.5	0.22	no	
6	15.0	0.27	ves	
7	17.5	0.32	yes	
8	7.5	0.13	yes	
9	5.0	0.09	no	
10	5.3	0.09	no	
11	5.6	0.10	no	
12	5.9	0.10	yes	
13	6.2	0.11	yes	
14	5.0	0.09	no	
15	5.3	0.09	no	
16	5.6	0.10	no	
17	5.9	0.10	yes	
18	6.2	0.11	yes	

Run Number:	360			
Comments:	Dry, no fluid.			
	Slips between 27.8 and 30.0 deg			
	Average = 28.9 c	deg / Slip Index	= 0.55	
Measurement	Slip Data	Slip Index	Did device slip?	
Number	(deg.)		(yes/no)	
1	25.0	0.47		
			no	
3	30.0	0.58 0.70		
3	35.0		yes	
5	37.5	0.77	yes	
6	25.0 27.5	0.47 0.52	no	
7		0.52	no	
	30.0 32.5		no	
8		0.64	yes	
9	27.5 27.8	0.52 0.53	no	
10	_		_	
- 11	28.1	0.53	yes	
	325.9	6.29	0.57220411	
	29.6	0.57		
		0.6		
		0.6		

Run Number:	358			
Comments:	EcoFlo Residue appears shiny & slick, no streaks			
	evenly distributed, similar to previous run.			
	CO2 cartridge ra		ooint.	
	Slips between 3			
	Average =3.3 de	g / Slip Index =	0.06	
Measurement	Slip Data	Slip Index	Did device slip?	
Number	(deg.)	•	(yes/no)	
	` •		,	
1	5.0	0.09	no	
2	5.3	0.09	yes	
3	5.6	0.10	yes	
4	5.9	0.10	yes	
5	5.0	0.09	yes	
6	2.5	0.04	no	
7	2.8	0.05	no	
8	3.1	0.05	yes	
9	3.4	0.06	yes	
10	2.5	0.04	no	
11	2.8	0.05	no	
12	3.1	0.05	no	
13	3.4	0.06	yes	
14	3.7	0.06	yes	

Run Number:	361		
Comments:	PG, residue visi	ble, fluid streaki	ng noted.
	Appeared to be slightly more residue at aft end		
	of test section.		
	Slips between 2	2.8 and 23.1 deg	
	Average = 23.0	deg / Slip Index	= 0.42
Measurement	Slip Data	Slip Index	Did device slip?
Number	(deg.)		(yes/no)
1	10.0	0.18	no
2	15.0	0.27	no
3	17.5	0.32	no
4	20.0	0.36	no
5	22.5	0.41	no
6	25.0	0.47	yes
7	27.5	0.52	yes
8	15.0	0.27	no
9	17.5	0.32	no
10		0.36	no
11	22.5	0.41	no
12	22.8	0.42	no
13		0.43	yes
14	23.4	0.43	yes
	281.8	5.17	0.369241025
	Į		

Run Number:	362		
Comments:	PG, residue visible, fluid streaking noted.		
	Last point (pt 15) measured on side of test section		
	Slips between 5		
	Average = 6.3 de	eg / Slip Index =	0.11
Measurement	Slip Data	Slip Index	Did device slip?
Number	(deg.)	•	(yes/no)
	` ,		,
1	10.0	0.18	no
2	15.0	0.27	no
3	20.0	0.36	yes
4	22.5	0.41	yes
5	10.0	0.18	yes
6	5.0	0.09	no
7	7.5	0.13	no
8	7.8	0.14	no
9	10.0	0.18	no
10	12.5	0.22	,
11	7.5	0.13	yes
12	5.0	0.09	no
13	7.5	0.13	yes
14	7.5	0.13	,
15	7.5	0.13	no

Run Number:	364		
Comments:	PG, residue visible, fluid streaking noted.		
	Repeat of Run 361.		
	Slips between 5	.0 and 5.6 deg	
	Average = 5.3 de	eg / Slip Index =	0.09
Measurement	Slip Data	Slip Index	Did device slip?
Number	(deg.)		(yes/no)
		2.12	
1	10.0	0.18	no
2	15.0	0.27	no
3	17.5	0.32	yes
4	10.0	0.18	yes
5	5.0	0.09	no
6	5.3	0.09	yes
7	5.6	0.10	yes
8	2.5	0.04	no
9	5.0	0.09	no
10	5.3	0.09	no
11	5.6	0.10	yes
12	7.5	0.13	yes
<u> </u>			

Run Number:	363			
Comments:	PG, residue visible, fluid streaking noted.			
	Slips between 5.6 and 7.5 deg			
	Average = 6.6 de	eg / Slip Index =	0.11	
Measurement	Slip Data	Slip Index	Did device slip?	
Number	(deg.)		(yes/no)	
	10.0	0.40		
1	10.0	0.18	no	
2	15.0	0.27	yes	
3	5.0	0.09	no	
4	7.5	0.13	no	
5	10.0	0.18	yes	
6	10.0	0.18	yes	
7 8	5.0 5.3	0.09 0.09	no no	
9	5.6	0.09	no	
10	7.5	0.10	ves	
10	7.8	0.13	yes	
12	8.1	0.14	yes	
12	0.1	0.14	yes	
	STDEV	0.05		
	SAMPLES	0.05		
		12.00		
	Avg.	0.14		
	-	.14 plus or minus	.03	
		,		

Run Number:	365			
Comments:	PG, residue visil	ble. fluid streaki	na noted.	
	Slips between 2.5 and 5.0 deg			
	Average = 3.8 de		0.07	
		-		
Measurement	Slip Data	Slip Index	Did device slip?	
Number	(deg.)		(yes/no)	
1	10.0	0.18	no	
2	15.0	0.27	yes	
3	17.5	0.32	yes	
4	5.0	0.09	yes	
5	2.5	0.04	yes	
6	0.0	0.00	no	
7	0.3	0.01	no	
8	0.6	0.01	no	
9	0.9	0.02	no	
10	2.5	0.04	no	
11	5.0	0.09	yes	
12	7.5	0.13	yes	

Run Number:	366			
Comments:	PG, residue visible, fluid streaking noted.			
	Slips between 2.5 and 2.8 deg			
	Average = 2.7 de	eg / Slip Index =	0.05	
Measurement	Slip Data	Slip Index	Did device slip?	
Number	(deg.)		(yes/no)	
1	10.0	0.18	no	
2	12.5	0.22	yes	
3	15.0	0.27	yes	
4	2.5	0.04	no	
5	5.0	0.09	yes	
6	0.0	0.00	no	
7	2.5	0.04	no	
8	2.8	0.05	yes	
9	3.1	0.05	yes	
10	3.4	0.06	yes	
11	2.5	0.04	no	
12	0.0	0.00	no	
13	2.8	0.05	yes	

368 Dry no fluid		
Slips between 28.1 and 28.4 deg Average = 28.3 deg / Slip Index = 0.54		
	Slip Index	Did device slip?
(deg.)		(yes/no)
		no
		yes
		yes
		yes
		no
		no no
_		no
		no
		yes
		yes
20.7	0.55	yes
	Dry, no fluid. Slips between 2	Dry, no fluid. Slips between 28.1 and 28.4 deg Average = 28.3 deg / Slip Index Slip Data (deg.) 20.0 0.36 22.5 0.41 25.0 0.47 27.5 0.52 30.0 0.58 32.5 0.64 35.0 0.70 20.0 0.36 25.0 0.47 27.5 0.52 27.8 0.53 28.1 0.53 28.1 0.53

Run Number:	367		
Comments:	PG, residue visible, fluid streaking noted.		
	Slips between 0.0 and 0.3 deg		
	Average = 0.2 de	eg / Slip Index =	0.003
Measurement	Slip Data	Slip Index	Did device slip?
Number	(deg.)		(yes/no)
1	10.0	0.18	yes
2	12.5	0.22	yes
3	0.0	0.00	no
4	0.5	0.01	yes
5	0.0	0.00	no
6	0.3	0.01	yes
7	0.3	0.01	yes
8	0.6	0.01	yes
9	0.0	0.00	no
10	0.3	0.01	yes

Run Number: Comments:	369 EcoFlo Residue appears shiny & slick, no streaks fluid evenly distributed. Slips between 2.5 and 3.1 deg Average = 2.8 deg / Slip Index = 0.05 Slip Data Slip Index Did device slip?		
Number	(deg.)		(yes/no)
	` 3,		,
1	10.0	0.18	no
2	15.0	0.27	no
3	20.0	0.36	yes
4	22.5	0.41	yes
5	10.0	0.18	yes
6	5.0	0.09	yes
7	0.0	0.00	no
8	2.5	0.04	no
9	2.8	0.05	no
10	3.1	0.05	no
11	5.0	0.09	no
12	5.3	0.09	yes
13	0.0	0.00	no
14		0.04	no
15	3.1	0.05	yes
16	5.0	0.09	yes

Run Number:	370		
Comments:	EcoFlo Residue	slick, no streaks	
	fluid evenly dist		
	Slips between 7	.5 and 7.8 deg	
	Average = 7.7 de	eg / Slip Index =	0.13
	_		
Measurement	Slip Data	Slip Index	Did device slip?
Number	(deg.)		(yes/no)
1	40.0	0.40	
	10.0	0.18	no
2	15.0	0.27	no
3	17.5	0.32	yes
5	20.0 7.5	0.36 0.13	
6	0.0	0.13	yes
7	2.5		no
8	5.0	0.04 0.09	no no
9	5.3	0.09	no
10	5.6	0.09	no
11	7.5	0.10	no
12	7.8	0.13	yes
13	8.1	0.14	yes
14	7.5	0.14	yes
17	7.5	0.10	ycs

Run Number:	372			
Comments:	EcoFlo Residue	appears shiny &	slick, no streaks	
	fluid evenly distributed.			
	Slips between 5	.0 and 5.3 deg		
	Average = 5.2 de	eg / Slip Index =	0.09	
B4	0!: D-1-	01: 11	Did decise die 0	
Measurement	Slip Data	Slip Index	Did device slip?	
Number	(deg.)		(yes/no)	
1	10.0	0.18	no	
2	12.5	0.18		
3	15.0	0.22	yes yes	
4	2.5	0.27	yes no	
5	5.0	0.04	no	
6	7.5	0.09	yes	
7	7.8	0.13	yes	
8	8.1	0.14	yes	
9	2.5	0.04	no	
10	2.8	0.05	no	
11	5.0	0.09	no	
12	5.3	0.09	ves	
13	5.6	0.10	yes	
14	2.5	0.04	yes	
15	0.0	0.00	no	
16	2.5	0.04	no	
17	5.0	0.09	no	

Run Number:	371			
Comments:	EcoFlo Residue appears shiny & slick, no streaks			
	fluid evenly dist			
	Slips between 2	.5 and 5.0 deg		
	Average = 3.8 de	eg / Slip Index =	0.07	
		<u></u>		
Measurement	Slip Data	Slip Index	Did device slip?	
Number	(deg.)		(yes/no)	
1	10.0	0.40		
2	10.0 15.0	0.18 0.27	no	
3	17.5	0.27	yes	
4	5.0	0.32	yes	
5	0.0	0.09	yes no	
6	2.5	0.00	no	
7	2.8	0.04	yes	
8	3.1	0.05	yes	
9	3.1	0.05	yes	
10	0.0	0.00	no	
11	2.5	0.04	no	
12	2.8	0.05	no	
13	3.1	0.05	no	
14	5.0	0.09	no	
15	5.3	0.09	yes	
16	5.6	0.10	yes	
17	2.5	0.04	yes	
18	2.5	0.04	yes	

Run Number: Comments:	273 EcoFlo Residue appears shiny & slick, no streaks fluid evenly distributed. Slips between 2.5 and 5.0 deg Average = 2.5 deg / Slip Index = 0.04 Slip Data Slip Index Did device slip?		
Number	(deg.)		(yes/no)
1	10.0	0.18	no
2	15.0	0.27	yes
3	17.5	0.32	yes
4	5.0	0.09	no
5	7.5	0.13	yes
6 7	2.5	0.04	yes
8	0.0	0.00 0.01	no
9	0.3	0.01	no
10	0.8	0.01	no no
11	2.5	0.02	
12	5.0	0.09	yes
13	7.5	0.13	yes
14		0.05	yes
15	2.5	0.04	yes
16	2.5	0.04	ves
			·

Run Number:	374		
Comments:	EcoFlo Residue a	appears shiny &	slick, no streak
	fluid evenly distributed. Slips between 2.5 and 3.1 deg Average = 2.8 deg / Slip Index =		
			0.05
Measurement	Slip Data	Slip Index	Did device slip
Number	(deg.)		(yes/no)
1	5.0	0.09	no
2	10.0	0.18	
3	12.5	0.22	yes
4	15.0	0.27	yes
5	17.5	0.32	yes
6		0.09	yes
7	0.0	0.00	no
8	2.5	0.04	no
9	2.8	0.05	, .
10	5.0	0.09	,
11	0.0	0.00	no
12		0.04	no
13		0.05	
14		0.05	, .
15	5.0	0.09	ye
	1		I

Run Number:	375				
Comments:	Dry, no fluid.				
	Slips between 25.3 and 25.6 deg Average = 25.5 deg / Slip Index = 0.48				
		<u> </u>			
Measurement	Slip Data	Slip Index	Did device slip		
Number	(deg.)		(yes/no)		
1		0.36	no		
2		0.41	no		
3		0.47	no		
4		0.52			
5		0.58	yes		
6		0.64	yes		
7	35.0	0.70	, .		
8		0.36	no		
9		0.47	ne		
10		0.47	no		
11		0.48			
12		0.49	yes		
13	30.0	0.58	yes		
	1		1		